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ALLOYING EFFECTS ON
TUNGSTEN-FIBER-REINFORCED
COPPER-ALLOY OR
HIGH-TEMPERATURE-
ALLOY MATRIX COMPOSITES

by Donald W. Petrasek and John W. Weeton
Lewis Research Center
Cleveland, Ohio

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SUMMARY

Relatively few metal-metal systems exist that would permit the creation of fiber-metal composites consisting of mutually insoluble constituents. It is anticipated that most high strength to weight ratio and high-temperature fiber composites ultimately to be produced will utilize high-strength fibers embedded in a highly alloyed matrix.

An investigation was conducted to determine the effect of alloying on the tensile properties and microstructure of tungsten-fiber-reinforced composites. Composites were made of tungsten fibers infiltrated with copper binary alloys that contain elements of varying solubility in tungsten. Room-temperature tensile tests were made on the composites, and a metallographic study of the microstructure of the fiber - metal-matrix interfaces was conducted. The tensile strengths and microstructures of these composites were compared with those of tungsten-fiber-reinforced - copper composites, which represent a mutually insoluble system. In addition, metallographic studies were made of tungsten fibers infiltrated with alloys, 70 percent nickel plus 30 percent iron, S-816, and L-605. Fibers also were chromium plated and infiltrated with copper.

Although all of the alloying additions of soluble elements made to copper-base binder materials lowered the strengths of the composites, some alloying elements did not do more than superficial damage to the fibers at temperatures at which other elements severely damaged the fibers. The degree of alloying with the fiber was shown to be a major factor influencing the tensile properties of the fiber and composite. It was shown that, for a given fiber content in a given binder system, as the depth of penetration of the alloy into the fiber increased, a decrease in both tensile strength and ductility of the composite resulted. A notch embrittlement effect due to the formation of a brittle alloy zone with the tungsten fiber was observed and gave rise to greater reductions in tensile properties and ductility behavior than would be predicted by a law-of-mixture relation alone.

INTRODUCTION

In recent years, investigators have considered combining fibrous materials with relatively weak binder materials. The interest in fiber-reinforced composites results from the fact that fibers or wires may be exceedingly strong and exhibit mechanical properties superior to those of the bulk materials from which they are derived. For example, tungsten wires drawn to less than 1 mil in diameter may have strengths of over 600,000 psi (ref. 1), while steel wires ranging from 10 to 3 mils in diameter may have strengths of the order of 300,000 to more than 600,000 psi (ref. 2). Both metallic and ceramic whiskers are known to have very high strengths. For example, iron whiskers have been shown to have strengths of 1.9 million psi (ref. 3), and sapphire whiskers have been reported to have strengths of the order of 1.7 million psi (ref. 4). If all or part of the strengths of such fibers could be retained subsequent to the incorporation of the fibers in an engineering material, a superior material could result.

In previous work done at the Lewis Research Center (ref. 5), the feasibility of fabricating fiber - metal-matrix composites that utilized the full strengths of the fiber and matrix materials was demonstrated. Tungsten fibers were combined with a copper matrix by a liquid-infiltration method such that the tungsten fibers were uniaxially oriented parallel to the tensile axis of the composite. A linear relation was established that related the tensile strength of the composite with the volume percent of the fibers. Mutually insoluble constituents were selected for the study so that the strength of the composite could be quantitatively related to the strength of the individual constituents.

The fact that there are only a few metals that are mutually insoluble with tungsten (e.g., copper, silver, gold, and zinc) and that these either have low melting points, high vapor pressures, or poor oxidation resistance makes it necessary to utilize other metals that are soluble in tungsten, where high-temperature applications are ultimately contemplated. It was anticipated that, for most practical fiber-reinforced composite materials, it would be necessary to use a fiber and a metallic matrix that would have varying degrees of solubility for each other. A logical extension of the earlier work was thus felt to be a study that would determine the effects of elements that had mutual solubility with tungsten.

Many factors may affect the strength of tungsten fibers, including effects from alloying elements; a review of several mechanisms by which tungsten-fiber properties are affected is thus necessary, so that alloying effects may be differentiated from concurrent effects due to other factors.

Tungsten fibers obtain a considerable portion of their strength from the mechanical working processes as the wires are being drawn to finer and finer diameters. Since highly worked materials recrystallize, there is a definite possibility that thermal treatments applied to the fiber might cause a reduction in the strength of the material by causing recrystallization of the fiber.

Numerous studies of the recrystallization behavior of tungsten wires have been made. For example, in reference 6, evidence has been shown that two types of recrystallization occur in tungsten wires. The first stage is described as a

low-temperature recrystallization that produces a columnar grain structure, and the second stage is a recrystallization at higher temperatures that produces a coarse-grained structure with a considerable reduction in strength. Wires that exhibit first-stage recrystallization are slightly ductile compared with those that have been recrystallized to the second stage of recrystallization. The bulk of the heat treatments or infiltration temperatures utilized in the study reported herein was in the region of the first stage of recrystallization for commercially drawn tungsten wires.

In addition to the damage that may be done to a tungsten fiber by thermal treatments, damage may also occur by the reaction or contamination of various alloying elements. Such elements as nickel, cobalt, and iron lower the recrystallization temperature of tungsten wires. Very small traces of nickel, as little as 10 angstroms thick on the surface of tungsten wires could cause an embrittlement of the wire on subsequent heat treatment (ref. 7). Contamination by nickel occurred from the practice of bending the wire around a nickel mandrel or by winding the wire over nickel rollers or onto nickel-alloy spools. Heat treatment subsequent to such practices caused a rapid diffusion of the nickel into the tungsten that resulted in a recrystallized, brittle wire. However, the nickel-contaminated wire was not embrittled at annealing temperatures below 1200° C. In a similar situation (ref. 8), it was found that as little as 1 percent of iron, nickel, or cobalt could embrittle tungsten wire. Wires that formed recrystallized regions on their surfaces as a result of the contamination and subsequent heat treatment fractured easily under stress and in a brittle manner. Cobalt was more damaging than either nickel or iron.

In reference 9, it was found that diffused nickel and chromium platings on molybdenum plus 0.5 percent titanium alloy rods caused the alloy to recrystallize. Recrystallization occurred at the periphery of the specimen only and caused a reduction in the mechanical properties and an embrittlement of the material. In the same study, it was also shown that an undiffused nickel plating did not cause a reduction in tensile strength of the fibers at temperatures up to 2000° F. Results of a similar nature might be expected for tungsten.

In addition to the damage to mechanical properties that might result from recrystallization by thermal treatments and diffusion of contaminants such as those mentioned previously, other types of damage have been reported. For example, carbon and ammonia have been shown to damage tungsten by embrittling the material during annealing treatments (refs. 10 and 11). This embrittlement results from diffusion of carbon and nitrogen into tungsten with the subsequent formation of brittle carbides and nitrides at the grain boundaries. Since tungsten has been shown to be notch sensitive, such an embrittling of the surface would be expected to be deleterious.

All the cases cited previously indicated that the tungsten fibers could be damaged by association with the different materials and thermal treatments described. However, the fracture strength of materials such as tungsten, particularly in wire form, may be improved by the removal of surface imperfections (thus improving the ductility) by procedures such as electropolishing. For example, improvements in the room-temperature tensile strength of tungsten wires of as much as 30 percent could be obtained by removing a small amount of material from

the surfaces of the wires (ref. 6). By electropolishing 0.1-inch-diameter tungsten rod, an increase of approximately 42 percent in the room-temperature strength (from 153,000 to 219,000 psi) could be obtained if the diameter of the rod was reduced 0.01 inch (ref. 12). In reference 13 the room-temperature strength of recrystallized 0.170-inch-diameter tungsten rod could be increased by approximately 20 percent (from 68,000 to 81,000 psi) as a result of removing 0.005 inch from the surface by electropolishing. In another investigation (ref. 14), it was found that heating tungsten rod in oxygen at 1500° F can enhance the ductility if the oxidation is allowed to continue for a sufficient period of time such that the roughened surface becomes smooth. The increase in ductility is believed to be due to the same mechanism that operates in electropolishing surfaces, namely, the removal of surface defects.

Numerous factors that may affect the strength of tungsten fibers have been presented. Relatively little work with composites that contain tungsten fibers has been reported. In reference 15, it is shown that kinked, short-length tungsten, steel, and molybdenum fibers could strengthen such matrices as silver, gold, and copper. Some of the combinations of fibers and matrices studied in reference 15 had some solubility with each other, while others were insoluble. The strength of several low-melting-point metals and alloys could be increased by reinforcing them with steel wool according to reference 16. In another study (ref. 17), a titanium alloy was reinforced with molybdenum fibers, and significant strength increases were obtained relative to the strength of the titanium alloy alone.

In these investigations, some of the fibers and matrices studied were soluble in each other; however, they were not designed to determine the effects of alloying or solubility on the strength of the fiber or the composite.

From these considerations, it can be seen that work is needed to determine the strengths of composites as they are related to alloying reactions between the fiber and matrix materials. This investigation, therefore, was conducted to obtain an understanding of some effects of alloying additions made to fiber-metal-reinforced composite matrices. The principal intention of the investigation was to determine some effects of differing alloying elements with differing solubility relations with tungsten on the tensile properties of tungsten-fiber-reinforced copper-alloy composites. In an effort to obtain an insight that ultimately will facilitate creation of high-temperature composites, a metallographic study was made of tungsten fibers infiltrated with several high-temperature alloys.

In this investigation, copper was utilized as a carrier medium, that is, a medium to which alloying elements soluble in tungsten were added and transported to the surfaces of the tungsten fibers (in the liquid state) during the fabrication practice. The utilization of copper-base alloys as infiltrants permitted a study of the reaction of the element of interest on the surface of the fiber. Evaluations of the reactions of alloying elements added to the copper carrier were made possible by determining the tensile strengths of composites made from the fibers and alloy infiltrants and by making microstructural studies of the fibers within the composites. The strengths of the composites were then compared with strengths of mutually insoluble tungsten-fiber-reinforced - copper composites.

Composites were made of tungsten fibers infiltrated with copper binary alloys that contain elements of varying solubility in tungsten; aluminum (Al), chromium (Cr), cobalt (Co), niobium (Nb), nickel (Ni), titanium (Ti), and zirconium (Zr). The volume percent fibers contained in the composites ranged from 65 to 80 percent. Compositions of the matrix infiltrants were selected so that alloy melting points were 2100° F or less. Specimens were made by liquid-phase infiltration techniques at 2200° F and for 1 hour in vacuum. On the basis of temperature and time alone then, fiber bundles could be made without recrystallizing the tungsten fibers. Metallographic studies were made of tungsten fibers infiltrated with alloys 70 percent nickel plus 30 percent iron, S-816, and L-605. Fibers also were chromium plated and infiltrated with copper.

MATERIALS, APPARATUS, AND PROCEDURE

Fibers

Commercially pure tungsten wires (General Electric type 218CS) 0.005 inch in diameter were selected as the reinforcing fiber material. The wires were observed to have a room-temperature tensile strength of 330,000 psi after an annealing treatment at 2200° F for 1 hour in vacuum. This is the same strength value reported in reference 5 after the wires had been given an identical annealing treatment. The fibers were given this treatment to determine the thermal damage to the tensile properties of the tungsten fibers when infiltrated with copper at these same conditions. The infiltration temperature used in this investigation is the same as that of reference 5, so that a direct comparison can be made with the results of that investigation.

It should be noted that the infiltration temperature lies in the region of the early stage of the first stage of recrystallization, as defined in reference 6. The "as-drawn" room-temperature strength of the wires was reduced from 390,000 to 330,000 psi by the thermal treatment used in this study. Accompanying this drop in strength was a widening of the fibrous grains of the tungsten wires. This change in microstructure, however, was only observable in longitudinal views of the wires and could not be detected in cross-sectional views. The term recrystallization used throughout this study refers to the formation of equiaxed grains, large enough to be observed optically in cross-sectional views of the tungsten wires.

Infiltrants or Binder Materials

Copper-base binary alloys that contain elements of varying solubility in tungsten were selected as matrix or binder materials, so that a comparison could be made with the results of reference 5 in which pure copper was used as a binder. Any change in properties of a tungsten-fiber-reinforced composite with copper alloys as binders relative to using copper as a binder will thus be due to the alloying additions to the copper. The weight percent of an element alloyed to copper was limited by melting-point considerations. As noted earlier, the temperature selected for infiltration was 2200° F. The amount of an element added to copper was thus selected so that the melting point of the copper alloy

was below 2100° F. The compositions of the copper binary alloys selected as binders are given in table I.

Specimen Fabrication

Bundles of 4-inch-long tungsten wires were cleaned to remove surface films and oxides by immersing in a hot, saturated solution of sodium peroxide and water, followed by rinsing in distilled water, immersing in a boiling solution of ammonia and water, and then rinsing again. The wires were then inserted into ceramic tubes so that the tube contained a range of volume percent of wire from 65 to 80. The ceramic tube was placed in a closed-end quartz tube with a slug of the binder material to be infiltrated on the bottom. The entire assembly was then placed in a resistance-heated vacuum furnace and heated at 2200° F for 1 hour.

The composites thus obtained were tungsten fibers bound by matrices that have varying degrees of solubility in tungsten. The specimens were 0.04 inch in diameter and contained continuous-length tungsten wires.

The specimens were then inserted into a 1/2-inch-deep hole drilled in a threaded rod. The specimens were brazed to the threaded rod with Aircosil number 45 (composed of 45 percent silver, 15 percent copper, 16 percent zinc, and 24 percent cadmium) as the brazing material. These specimens were then tested in tension at room temperature.

Test Procedure

Room-temperature tensile tests were made on the copper-alloy - tungsten-fiber composites with an Instron tensile machine (screw-driven) at a constant crosshead speed of 0.01 inch per minute. A strip-chart recording of load against displacement was made for each test.

Reduction in area at the fracture edge of the composites was determined by measurements made with a comparator at a magnification of 100.

The procedure used for determining the relative amount of fiber or binder was to section the specimen after testing and then measure the cross-sectional area by planimetering a photograph of the cross section. This area was used as the basis for tensile-strength calculations. A wire count was made of the cross section to determine the volume percent of the fiber present.

Metallographic Studies

Metallographic studies were made of the cross sections of the tungsten-fiber-reinforced copper-alloy composites. All the specimens were swab etched with a solution of five parts ammonium hydroxide plus one part hydrogen peroxide to reveal the copper-alloy structure, and Murakami's etchant (10 percent potassium hydroxide, 10 percent potassium ferricyanide, and 100 cubic centimeters

of water) to reveal the structure of the tungsten wires. Photomicrographs were then taken at magnifications of 250 and 750.

Measurements of the depth of visible penetration or depth of alloying of the binders with the tungsten fibers were made on the etched cross sections of the composites at a magnification of 750 with a Filar eyepiece.

Electron photomicrographs were also taken of the cross section of some selected composites, where views at higher magnifications were felt to be of interest. Parlodion replicas were made from specimens shadowed with chromium at an angle of 30° .

Miscellaneous Alloying Additions and Approaches

Electroplating with chromium. - It was desired to determine the effects of high chromium additions on the microstructure of tungsten fibers. However, copper that contains high concentrations of chromium above 2 percent (the maximum quantity indicated in table I) could not be used because of melting-point considerations, since higher chromium additions raise the melting point of the copper alloy above that which could be used to infiltrate tungsten fibers successfully without causing the fibers to recrystallize. For this reason, tungsten fibers were chromium plated and annealed at 2200° F for 1 hour to determine the effect of chromium plating on the microstructure of tungsten fibers. The plated wires were given an intermediate annealing treatment at 500° F for 1 hour to remove occluded hydrogen from the deposit. Bundles of chromium-plated tungsten wires were also subsequently infiltrated with copper to determine if the plate would remain intact after infiltration, since it was of interest to determine the feasibility of plating fibers with materials that could act as diffusion barriers to alloying of the fibers by the matrix material.

Inhibition of recrystallization by a "buffering" addition. - Early in the investigation it was observed that certain alloying additions to copper caused the tungsten fibers to recrystallize. Nickel was one of the recrystallization-promoting elements. To determine if the promotion of recrystallization by nickel could be buffered or stopped, an alloy containing 52 percent niobium and 48 percent nickel was made and used as a binder material. It was hypothesized that, by saturating the nickel with another refractory metal, the alloying of nickel with tungsten fibers might be hindered. Composites were thus made with this alloy as a binder. The composites were made at 2200° F for 1 hour in vacuum by the same technique used to make the copper-alloy composites. A microstructural study was then made of these specimens.

Utilization of a nickel-iron alloy as a binder material. - To investigate the effect of higher percentages of known contaminants of tungsten wire, a nickel-base alloy containing 30 percent iron was used as a binder material. The composite was made by liquid-phase infiltration at 2800° F for $1/2$ hour in vacuum. A microstructural study was then made of these specimens.

Commercial alloys used as binder materials. - Commercial cobalt-base alloys with good high-temperature strength properties and good corrosion-resisting

qualities were also used as binders. Composites consisting of L-605 and S-816 as binders were made by liquid-phase infiltration at 2700° F for 1 hour in vacuum. A microstructural study was made of these specimens. The composition of these alloys is given in table II.

RESULTS

Tensile Tests

Room-temperature tensile data plots for the copper-alloy - tungsten-fiber composites are shown in figure 1. The solid curve on each plot (a base line for comparison) represents the linear relation that was obtained in reference 5 between the tensile strength of tungsten-fiber-reinforced pure copper-matrix composites and the volume percent of the fibers in the composite or which might be calculated knowing the strength of the copper and tungsten fiber.

Table I compares the tensile strength, volume percent fiber content, reduction in area, and type of fracture of the copper-alloy - tungsten-fiber composite. It is evident that some copper alloy, namely, 5 percent nickel, 10 percent titanium, 10 percent zirconium, 1 and 2 percent chromium, and 1 percent niobium, did not seriously affect the tensile properties of the composite. On the other hand, composites consisting of 10 percent nickel, 5 percent cobalt, 5 and 10 percent aluminum, 25 percent titanium, and 33 percent zirconium additions to copper had a substantially lower tensile-strength value than did the specimens bonded by pure copper.

Fracture Ductility

The reduction in area of the composites as given in table I was determined macroscopically by observing the fracture edges. It was difficult to obtain an exact value for the reduced area at the fracture edge because of the jagged fracture and wire "pull-out" at fracture. The reduction-in-area data obtained, however, indicate qualitatively the relative degree of ductility of the composites. It may be observed generally that those specimens that had the greatest degree of ductility at fracture exhibited the highest tensile-strength values. The photomicrographs (fig. 2) show typical fracture edges of the composites. It should be noted that a measurable ductility can result from either the necking down of the composite at the fracture edge, as is evident in figure 2(h), or the necking down of individual fibers, as is the case in figure 2(i).

Load-Deformation Curves

Load-deformation curves were obtained for each specimen tested. Such deformation curves give a qualitative indication of the elongation against load rather than a stress-strain curve, since they are made by measurements of the motion of the crosshead of the tensile machine. On the other hand, the shape of the curve indicates the degree of ductility that is observable at the fracture edge of the specimen, and, in fact, deformation curves were used to categorize

fractures such as brittle, semiductile, and ductile. Figure 3 shows such curves for the copper 5 percent cobalt - tungsten-fiber composites, and examples of the types of deformation are indicated in the figure.

Microstructural Studies

Copper - tungsten-fiber specimens. - Microstructures of a pure copper - tungsten-fiber specimen (ref. 5) are shown in figures 4 and 5 to furnish a base line for comparison. Figures 4 and 5 show that recrystallization of the fibers has not taken place and no phase or alloying is seen around the fibers, which would be expected since tungsten and copper are mutually insoluble.

Copper-nickel - tungsten-fiber specimens. - It is evident from figure 6 that the 5 percent nickel matrix did not produce any visible recrystallization at the metal-matrix - fiber interface. As discussed later, one specimen had three wires out of 40 in a cross section that were recrystallized. In the case of the specimens that contain 10 percent nickel in copper (figs. 7 and 8), recrystallization was evident in all of the fibers. It is interesting to note that rounded grains are formed at the periphery of the tungsten fibers (see fig. 8). These may have resulted from the dissolving and reprecipitation of tungsten by the nickel. The greatest depth of recrystallization in these 10 percent nickel specimens has been of the order of 0.0008 inch. It is believed that this zone consists of an area containing nickel that has diffused into the tungsten. It is also believed that the material recrystallized subsequent to or during this process. Approximate diffusion calculations indicate that a depth of penetration such as that observed is feasible under the time and temperature conditions used in the infiltration process.

Copper-cobalt - tungsten-fiber specimens. - In both of the microstructures shown (figs. 9 and 10), it is evident that there is some recrystallization near the surface of the fibers. At the actual surface of the fibers, there is an apparent border that is unrecrystallized. Thus, the recrystallization, in effect, is a subsurface recrystallization. This phenomenon is very apparent in figure 10 for the 5 percent cobalt specimen. In some areas of the specimen, a phase appeared around the fiber. This phase extended outside of the original diameter of the fiber. The recrystallization in the cobalt-containing matrices is different from the nickel-containing matrices not only in that subsurface recrystallization was produced but that the recrystallization had occurred with the formation of smaller grains. On the other hand, it should be recalled that the percentages of cobalt that lead to recrystallization of the tungsten fibers are much less than those for the nickel. In fact, 5 percent nickel additions to copper did not produce any visible recrystallization of the tungsten fibers except for the case noted.

Copper-aluminum - tungsten-fiber specimens. - Figures 11 and 12 show photomicrographs of cross sections of copper-aluminum - tungsten-fiber composites. The specimens shown consist of tungsten fibers embedded in a copper 5 and 10 percent aluminum matrix, respectively. For these specimens, the aluminum in the matrix and in the diffusion zones produced etching complications. Thus, the recrystallized zone does not stand out as graphically as it does in the nickel

or cobalt specimens. However, a recrystallized zone is certainly evident for both the 5 and 10 percent aluminum specimens. For both of these specimens, the recrystallized zones are thicker than for the nickel and cobalt specimens. The observation of a dendritic structure in the copper 5 percent aluminum matrix and a eutectic structure in the copper 10 percent aluminum matrix is in good agreement with the copper-aluminum phase diagram (ref. 18), which contains a eutectic at copper $8\frac{1}{2}$ percent aluminum and 1900° F.

Copper-titanium - tungsten-fiber specimens. - Figures 13 and 14 show the cross section of specimens consisting of tungsten fibers embedded in a copper 10 percent titanium matrix. There is no evidence of recrystallization in these specimens. At the higher magnification (X750) in figure 13, a slight two-phase band seems to exist at the matrix-fiber interface that is probably a two-phase eutectoid type of zone not resolved by these photomicrographs. The matrix seems to have a considerable quantity of intermetallic compounds or precipitates. Figure 14 clearly shows that alloying of the surface of the fiber has taken place. The two-phase zone is resolved.

Figure 15 shows that the higher titanium addition to copper (25 percent) also did not cause any recrystallization of the fibers. Thus, the tendency observed in other systems to produce recrystallization at higher concentrations rather than at lower ones was not observed in this system. The higher titanium addition to copper, however, resulted in a much larger alloyed fiber zone.

Copper-zirconium - tungsten-fiber specimens. - Figures 16 and 17 show cross-sectional microstructures of a tungsten-fiber-reinforced - copper plus 10 percent zirconium alloy. There is no evidence of a recrystallization zone at the periphery of the fiber, but there is evidence, as seen from the electron photomicrograph (fig. 17), that slight alloying with the fiber has occurred. A eutectic type of structure is evident in the matrix or bonding material. The photomicrograph shows needles of an intermetallic compound, which probably is $ZrCu_3$. Figure 18 shows the microstructure of the 33 percent zirconium addition to copper. The photomicrograph shows almost no additional reaction at the metal-matrix - fiber interface compared with the lower percent addition of zirconium to copper, although the black band at the periphery is more pronounced. It should be noted that the matrix is brittle and consists largely of $ZrCu_3$.

Copper-chromium - tungsten-fiber specimens. - Figures 19 and 20 do not show any evidence of recrystallization in these specimens. A layer of what appears to be a single phase tungsten-rich structure was observed on the surface of the fiber.

Copper-niobium - tungsten-fiber specimens. - The photomicrograph in figure 21 does not show any evidence of recrystallization in the fibers. At the metal-matrix - tungsten-fiber interface is a darkly etched band that could conceivably be a diffusion zone where the niobium and the tungsten combine. The fact that a band exists in this case is a result of an etching effect.

Miscellaneous Alloying Additions and Approaches

Electroplating with chromium. - Figure 22(a) shows the microstructure of a chromium-plated tungsten fiber. The chromium is seen to be bonded to the tungsten fiber. The plated and annealed wires were then liquid-phase infiltrated with pure copper at 2200° F for 1 hour to see if the plate would still be present when attacked by a liquid metal of low solubility. It is clear from figures 22(b) and (c) that some of the plate is still present and thus might act as a barrier to alloying by other elements soluble in tungsten. It is evident that the chromium plating is very thick and is not the best quality plating possible. Nevertheless, it is apparent that the plating has not been completely dissolved by the infiltration process. As seen in the cross-sectional view of the copper-infiltrated specimen (fig. 22(c)), the time and temperature used caused the chromium to penetrate into the tungsten. This is revealed by the etched edge that has a different luster from that of the interior of the fiber.

Inhibition of recrystallization by "buffering" agent. - Results of the buffering experiment with niobium additions to prevent an attack or a recrystallization reaction by nickel on tungsten fibers is shown in figure 23. The black border at the edge of the fiber is an etching effect that probably results from the fact that two phases are present. Where nickel in copper causes a severe recrystallization of the fibers and where nickel alone as a pure metal is known from other experiments to produce the same effect, the nickel contained in the niobium did not produce a recrystallized zone at the periphery of the fiber. Thus, it may be possible to "buffer" a recrystallization reaction at the fiber-matrix interface by the addition of a material compatible with the fiber material. It is evident in the photomicrographs that the wires have been cracked by the process. The matrix material, in this case, is a eutectic that is relatively brittle, forming a Ni_3Nb intermetallic compound along with another intermetallic phase NiNb (ref. 18). Thus, the matrix is brittle enough to have numerous cracks in it, and these cracks seem to have propagated into and through the tungsten fibers. It should be noted here that severing of the fibers, as is observed in figure 23, has also been observed for some of the other alloying addition systems where recrystallization has not occurred. Splitting of the fibers is believed to have occurred during the cutting procedure of fabrication, and possibly, even in some cases, by a reaction analogous to a stress-corrosion phenomenon. In any case, the ease with which a fiber may be fractured in a brittle manner is evident from such a photomicrograph as that of figure 23. It would indicate certainly an inherent notch sensitivity of the fiber.

Nickel-iron alloy utilized as a binder material for tungsten fibers. - Figure 24 shows the microstructures of tungsten fibers infiltrated with a nickel-iron alloy containing 70 percent nickel and 30 percent iron. The infiltration temperature, 2800° F, of necessity, was considerably higher than the temperatures used for the copper-alloy system, since the melting point of the alloy was higher. The low-magnification photomicrograph of figure 24 shows that the fibers have essentially been thoroughly recrystallized, whereas the high-magnification picture shows that the actual recrystallized grains have, in effect, been separated by penetration of the iron-nickel alloy. Tungsten has been dissolved and reprecipitated to form large rounded grains by a reaction similar to that occurring in "liquid-phase sintering." The nickel-iron alloy, furthermore, has

dissolved a considerable amount of tungsten and formed an iron-nickel-tungsten structure that has a eutectoid appearance.

Tungsten wires bonded by commercial alloys. - Figure 25 shows the microstructures of tungsten wires infiltrated with S-816, and figure 26 shows the microstructure of tungsten wires infiltrated with L-605. The S-816 infiltrant produced a peripheral phase about the tungsten fibers. Some of the wires have not been fully recrystallized as is evident in figure 25, and the alloy has dissolved a considerable portion of the fibers. In fact, some fibers have been almost thoroughly dissolved. The microconstituents in the matrix are undoubtedly tungsten-rich. The infiltration temperature, of necessity, was higher than that used for the copper system, namely, 2700° F, which in itself would cause the fibers to recrystallize.

The L-605 infiltrant also dissolves a considerable portion of the fibers and recrystallization occurs. The inner portions of some of the fibers still seem to remain intact, however. In this case, again, a phase is noted on the periphery of the fibers and comprises a large portion of the matrix material. This matrix also is an unusually brittle one.

Penetration Measurements

Table III lists the depth of penetration of alloys or other types of reaction that occurred at the metal-matrix - fiber interface and gives a quantitative indication of the extent of the attack or reaction. It is apparent from table III that the copper-aluminum alloys penetrated to a greater depth than did any of the other copper alloys investigated. It is also apparent from the table that higher concentrations of a given element addition to copper gave increasing degrees of penetrations.

DISCUSSION

Tensile Strength Comparisons and Effect of Alloy Composition on Copper-Alloy - Tungsten-Fiber Composites Relative to

Copper - Tungsten-Fiber Composites

It was pointed out earlier that the tungsten fibers used in this investigation and in the investigation of reference 5 were given the same thermal treatment and thus had the same initial tensile strengths. Any reduction of tensile strength from the copper - tungsten-fiber composite base line thus represents the metallurgical damage done to the fibers or composites by the alloying additions to the binder materials. It was noted earlier that any combination of thermal and alloying treatments might more readily damage, rather than improve, the properties of the highly worked tungsten fibers. The mechanical properties of the composites tested in this study were reduced. It was, however, encouraging to find that several of the composites tested showed little reduction in tensile strength relative to the composites made from the insoluble materials, copper

and tungsten, that were used as a base line for comparison. Thus, highly worked fibers may be combined with selected alloying elements without severely reducing their properties. The average decrease and percent reduction in tensile strength of these composites are given in table IV.

It should be noted from the values given in table IV that the copper 33 percent zirconium and copper 25 percent titanium binders resulted in a large reduction in tensile strength. These composites were fabricated and tested to augment what appeared to be a possible alloying behavior noted in early stages of the investigation. It was observed that low percentages of nickel additions to copper did not cause a recrystallization zone around the periphery of the tungsten fibers, whereas high percentages of nickel additions to copper did. It was also observed that additions to copper of 10 percent zirconium or 10 percent titanium did not cause recrystallization zones to form. Since zirconium and titanium could be added to copper in large percentages without raising the melting point of the binder alloy, it was worthwhile to determine whether large percentages of these elements would also cause recrystallization. The metallographic results showed that these large alloy additions did not cause recrystallization although a larger alloyed zone was produced. The fact that these binders did significantly lower the tensile strength of the composites relative to the tensile strength of pure copper - tungsten-fiber-reinforced composites was almost certainly due to the brittle nature of the matrix material itself. In the case of the 33 percent zirconium binder material, the matrix was almost entirely a brittle intermetallic; and, in the case of the 25 percent titanium binder, large quantities of intermetallics were present. A discussion of the mechanism of damage to the tensile properties of the composite in these special cases will be made later. Thus, comparisons of damage to the fibers by alloying reactions with the fibers should be made only with the lower titanium and zirconium additions to copper (e.g., 10 percent zirconium, 10 percent titanium), which did not contain large quantities of intermetallic compounds.

The values in table IV, with the exception of these two binders, thus clearly show that the greatest damage by alloying reactions with the fiber was done by high percentages of nickel and by aluminum or cobalt additions to copper, and that much less damage was done by additions of titanium, chromium, zirconium, niobium, and low percentages of nickel.

For a given alloy matrix system with an increasing alloy content to the binder material, there was generally an increasing degree of attack, or damage, to the composite structure, as is revealed in table IV.

Table V shows the phase equilibrium relations of the alloying elements added to copper with tungsten. It appears from the values of tables IV and V that the body-centered cubic materials did far less damage to the tungsten fibers than did the face-centered cubic materials. Of course, the fact that most of the body-centered cubic materials have melting points closer to that of tungsten, as well as the fact that they are isomorphous with tungsten, suggests that these materials should be more compatible with tungsten and that they should have a lesser effect on the properties of tungsten. Furthermore, it is suggested that the depth of penetration and the degree of recrystallization would be less for materials that are relatively compatible with or similar to tungsten.

It can also be seen from tables IV and V that the alloying elements that did the most damage to the properties of the fibers in the composites, namely, nickel, aluminum, and cobalt, had low solubilities in tungsten. Their atomic radii are also widely different from that of the tungsten relative to most of the other materials investigated. This should indicate that these materials would have a more rapid diffusion rate into the tungsten than body-centered cubic materials with atomic radii close to that of tungsten. The combined damage that the face-centered cubic materials seem to have done to the tungsten fibers is believed to involve diffusion into the material and subsequent recrystallization of the tungsten. It may also be noted that the solid solubility of tungsten in the elements studied is, in many cases, very large. This includes the cases of the materials containing cobalt and nickel plus other elements that did not do much damage. The fact that the greatest damage to the composites was done by those materials where diffusion and recrystallization took place within the tungsten suggests that the solid solubility of tungsten in the binder materials is not as important a factor to consider as the solubility of the alloying elements in tungsten.

Correlation of Tensile Properties of Copper-Alloy - Tungsten-Fiber Composites with Microstructure

Three types of phenomena were observed to occur at the metal-matrix - tungsten-fiber interface:

(1) A diffusion-penetration reaction accompanied by a recrystallization of the grains at the periphery of the tungsten fiber occurred. Such a reaction is very evident for the specimens containing cobalt, aluminum, and 10 percent nickel, as shown in figures 7, 9, 10, 11, and 12.

(2) A two-phase zone was present. The two materials that produced such structures were the copper-titanium and copper-zirconium binder materials. At the temperature utilized for the infiltration process, 2200° F, it is believed that the alloying additives to copper diffused into the tungsten and subsequently caused the formation of a phase. In the case of the copper-zirconium alloys, it is believed that the phase formed was a ZrW_2 phase. In the case of the titanium-bearing infiltrants, the reaction that is believed to have occurred produced a eutectoid. Such observations are based entirely on metallographic examinations including those shown at high magnification in figure 14 along with the use of the equilibrium diagrams of reference 18.

(3) A solid-solution reaction without subsequent recrystallization took place. This type of interface reaction is largely that of a solid-solution reaction between tungsten and the additive without any evidence of surface recrystallization. It was observed in the case of the copper-niobium and copper-chromium alloys.

The type of reaction that occurred at the copper-alloy tungsten-fiber interface is given for every binder material investigated in table III. The results clearly show that the most damaging type of reaction at the interface was the

diffusion-penetration reaction accompanied by recrystallization. This type of reaction occurred with the copper plus 10 percent nickel, copper plus 1 and 5 percent cobalt, and copper plus 5 and 10 percent aluminum binder materials. Solid-solution reactions without subsequent recrystallization did not seriously affect the tensile properties of the composites. The formation of a two-phase zone at the interface did not result in as great a reduction in tensile properties of the composites as did the diffusion-penetration reaction accompanied by the recrystallization reaction. It is interesting to note from table IV that the copper plus 25 percent titanium binder, which formed a two-phase zone at the interface and which penetrated the fiber to a much greater depth than did either the copper plus 10 percent nickel or copper plus 5 percent cobalt binders, reduced the tensile strength of the composite 27 percent. However, the copper plus 10 percent nickel binder and the copper plus 5 percent cobalt binder reduced the tensile strengths of the composites by 62 and 24 percent, respectively. The copper plus 33 percent zirconium composite had a two-phase reaction zone at the interface and resulted in reducing the tensile strength of the composite by 57 percent.

As mentioned previously, another mechanism by which the strength of the composite may be damaged is associated with the ductility behavior of the matrix. It was indicated that some of the copper alloys are brittle because of the intermetallic content of the matrix. In the case of the copper plus 33 percent zirconium binder, cracks formed in the matrix and extended into the fibers. In addition, some of the fibers split in two. This splitting is believed to be the result of a stress-corrosion phenomenon.

Correlation of Depth of Penetration With Tensile Strength and Ductility Behavior

Depth-of-penetration measurements of the recrystallized-diffusion zones in the copper plus 5 percent cobalt - tungsten-fiber composites are given in table VI. It can be seen that the depth of this zone varies even though all the specimens were given the same thermal treatment. It is known, however, from work of a preliminary nature done at Lewis, that even a single strand of wire varies in its resistance to recrystallization at a given temperature. Thus, it is not surprising that varying degrees of recrystallization could take place by alloying. In measuring the depth of penetration of the recrystallization zones for the copper plus 5 percent cobalt binders associated with the tungsten-fiber composites, it was observed that, as the depth of penetration of the recrystallized zone increased, there was a steady decrease in the tensile strength of the composite. The volume percent of fiber present in the composites is also approximately equal, as is noted in table VI. Thus, for a given fiber content, it was observed that, as the depth of penetration increased, a corresponding decrease in tensile strength was also observed. A decrease in the tensile strength of a composite due to alloying the fiber might be expected, since by alloying the fibers the true volume percentage of worked fiber in the composite essentially has been lowered. To obtain the same tensile strength as would have resulted if no alloying had occurred, the alloyed zone of the fiber must have the same tensile strength as the unalloyed portion. It is highly unlikely that the alloyed

zone would have such a high tensile strength. The results of this investigation bear this fact out. The degree of alloying with the fiber thus would determine the tensile properties of the composite: the greater the penetration, the greater the reduction in properties.

It was observed in a series of specimens that has approximately the same volume percent fibers that the tensile strength and ductility varied with the depth of the recrystallized zone. To understand better the ductility behavior and the tensile strength reductions associated with recrystallization depths in the fibers, two approaches were taken. The first shows that the reduction in the properties of the cobalt specimens, as well as in some of the other systems, is not solely the result of a geometric effect (law-of-mixtures relation) associated with the area occupied by the recrystallized zone of the fiber. The composite should be considered as a three-component system that consists of the copper-cobalt binder, the unrecrystallized tungsten wire core, and the recrystallized wire zone. If the strength of the recrystallized zone were known, the tensile strength of the three-component composite could be determined by using an equation similar to that presented in reference 5 and by using the methods described in the appendix. The strength of the recrystallized zone was not known, and therefore another approach was utilized. This approach is described in detail in the appendix and does not require a knowledge of the strength of the recrystallized zone. An equation was obtained that relates the depth of the recrystallization-penetration zone with the loss in properties of a tungsten-fiber-reinforced - copper-alloy composite relative to a tungsten-fiber-reinforced - copper composite. This is shown as follows:

$$\Delta\sigma_c = KA_f(2Pd_o - P^2)$$

where

$\Delta\sigma_c$ difference in tensile strength between tungsten-fiber - copper composite and tungsten-fiber - copper-alloy composite

A_f area percent fiber in cross section of a composite

$P/2$ total depth of penetration or depth of recrystallized zone (see sketch p. 24)

d_o initial diameter of fiber

Thus, K should be constant for a given alloy system if the penetration zone does not disproportionately damage the core of the fiber. To understand better the physical meaning of K , consider the relation (eq. (A4)) derived in the appendix for K : $K = (\sigma_f - \sigma_p)/d_o^2$. Obviously, on the basis of the assumptions needed to derive this equation, each term in the equation must be a constant. This has been done for the various systems, and the values of K are given in table VII. It is evident in the 5 percent cobalt binder system that there is a wide range of values of K , and thus the damage done to the composites is not solely related geometrically to the area occupied by the recrystallized zone. This is also true for binder materials such as copper-nickel and copper-zirconium. Such a calculation would probably be significant for only those

systems where three or more data points were obtained. In fact, it would be desirable to have many more data points for each system, but it was not the primary objective of this paper to prove this point. Consider then only the 5 percent cobalt, 5 percent nickel, 10 percent nickel, and 10 percent zirconium systems for which several data points exist. In all of these systems, a disproportionate amount of damage to the tensile strength properties of the composites was observed since K varied. It is also of interest to note that the fractures of the specimens that show the least reduction in strength were generally very ductile, whereas brittle fractures occurred for those specimens that had the greatest reductions in tensile strength.

The relation between ductility and tensile strength of the individual tungsten fibers in a copper plus 5 percent cobalt system and the depth of the recrystallization-penetration zone can be determined by a graphical approach. The tensile-strength contribution of the fiber (the fiber in this case includes the recrystallized zone) to the tensile strength of the composite can be obtained by finding the value of the 100 percent tungsten-fiber content intercept on a plot of tensile strength against fiber content for the composites tested (see fig. 27). Assume that the strength contribution of the copper alloys to the strength of the composite is the same as that obtained with pure copper as a binder. The tensile-strength contribution of the tungsten fiber to the tensile strength of the composite can be obtained by drawing a line from the pure copper - tungsten-fiber line intercept at zero percent fiber content A through the copper plus 5 percent cobalt system data point B to its intercept at 100 percent fiber content C. The 100 percent fiber content intercept thus gives the value of the tensile-strength contribution of each individual tungsten fiber. This tensile-strength value is then plotted against the percent area of the fiber that is recrystallized, as is shown in figure 28. The tensile-strength value for an unrecrystallized tungsten fiber used in this study is known to be 330,000 psi. An approximation of the tensile strength of the recrystallized zone of the fibers or of a fully recrystallized fiber may be obtained by drawing a straight line between the tensile strength of the unrecrystallized tungsten fiber A and the tensile strength of the tungsten fibers in the best copper plus 5 percent cobalt specimen B. This is believed to be a warranted approach since the best composite data point almost falls on the law-of-mixture curve for the copper - tungsten-fiber system (as shown in fig. 27). In addition, the fracture was a ductile one, as mentioned earlier. Point C in figure 28 is, therefore, an approximate value for the strength of the recrystallized fiber zone. The type of fracture observed for each specimen tested is also indicated in figure 28.

If the system obeyed a law of mixtures, all the data points for the copper plus 5 percent cobalt system should lie on the curve ABC. It is observed, however, that this is not the case. As the depth of penetration increased, an increased deviation in tensile strength from that predicted from a law-of-mixture consideration is observed. The ductility behavior is also seen to vary with depth of penetration. As the depth of penetration increases, ductility decreases. It is also of interest to note from figure 28 that a drastic deviation from the law-of-mixture curve does not occur until the fiber has been penetrated to a depth greater than approximately 0.0004 inch or until 30 percent of the fiber has been recrystallized.

In the case of composites with the copper plus 5 percent cobalt binders, it was also observed that, as the depth of the recrystallization-penetration zone increased, ductility decreased, as evidenced from the load against displacement curves shown in figure 3. A decrease in tensile strength with increasing depth of penetration was also observed. It is believed that the decrease in both tensile strength and ductility of the tungsten fibers and composites is a result of a notch-embrittlement effect. Tungsten is known to be notch sensitive. The fact that greater penetration depths resulted in lowered ductility is compatible with concepts of notch sensitivity. For example, in reference 19, in which the effects of brittle skins on otherwise ductile metals were determined, it was found that the ductility behavior of a metal under the influence of various case depths followed virtually the same pattern as that of the metal (without a brittle skin) under the influence of various notch depths. As the penetration depth increased, the ductility decreased. This then explains why a brittle skin decreases the ductility and tensile strength of a metal more rapidly than would be expected from only the consideration of the area occupied by the skin.

Fully recrystallized tungsten wire is extremely brittle at room temperature. The recrystallized zone of the fiber in composites that contain a copper plus 5 percent cobalt binder thus acts as a brittle skin. It is believed that the brittle recrystallized tungsten zone is the equivalent of a circumferential notch in the wire, which produces a notch-embrittlement effect.

Several specimens of the 10 percent nickel series had recrystallization-penetration zones comparable to those of the 5 percent cobalt series. The copper-nickel specimens were damaged to a much greater degree than were the copper-cobalt specimens (62 against 24 percent - see table IV). A larger recrystallized grain size and a greater concentration of alloying element in the tungsten present two possible explanations for this effect. As would be expected, a notch-embrittlement effect also occurred in this system.

In the case of a group of specimens that contain 5 percent nickel, it was observed that specimen 2, which had a reduction in tensile strength of 10,000 psi, however, had, within experimental measurement capabilities, the same depth of penetration as specimen 3, which had a larger amount of damage. It was observed, however, that three wires in specimen 3 had a recrystallized zone, a fact that could account for the lower properties of this specimen.

In the case of the 10 percent zirconium specimens, tabulated in table VII, the reaction at the interface is one that does not result in a recrystallized fiber zone; however, a nonuniform amount of damage results since the values of K vary considerably. The fact that the binder material consists, in part, of the intermetallic $ZrCu_3$ is believed to contribute to the disproportionate amount of damage observed. It was observed that the copper plus 33 percent zirconium - tungsten-fiber composite had a tensile strength that was only 50 percent of that of a copper plus 10 percent zirconium - tungsten-fiber composite. The depth of penetration, however, was not appreciably greater in the higher zirconium content composite. It should be noted that this brittle intermetallic phase could prohibit the full utilization of the strength of the fibers, since the matrix could fail before the fibers reached their ultimate strength. If the matrix fractures at an elongation that is less than the elongation at which the tungsten fibers

fracture, then it is apparent that the matrix will fail first and result in the tungsten fibers carrying more than their proportionate share of the load.

It appears, from what has preceded and from values given by table III, that all the specimens that have recrystallization-penetration zones greater than 0.0004 inch result in disproportionate damage to the properties of the fibers and that, in part, this is due to a notch sensitivity of the fiber. In summary, it has been shown, on an overall basis, that those specimens with the greatest ductility had the highest strengths, that generally those specimens with the brittle fractures are representative of notch-sensitive materials, and that this is particularly true with those materials that have been infiltrated with alloy additions that cause a diffusion-penetration reaction accompanied by recrystallization at the edge of the fiber. It is also felt that the greater the depth of such diffusion-penetration zones, the greater the tendency in a given system for the material to become more brittle. It is evident that, if a comparison is made between low percent alloying additives made to the copper-base binder materials and high alloy percent additive binders, such as the cobalt- and the nickel-bearing binder materials, the embrittling effect is greater for a given amount of penetration for the binder that contains the larger percentages of elements, which causes the embrittling effect.

Tungsten Fibers - Higher-Melting-Point Alloys

One of the observations made of the microstructure of the tungsten fiber infiltrated with high-melting-point alloys was that a gross dissolving of the fibers took place. This type of reaction does not apply to the specimens that were associated with the copper-base binder materials. It was only observed where high-melting-point materials were infiltrated at higher temperatures than the copper-alloy binder systems. Included were the effects of S-816, L-605, and the nickel-iron alloys on the fibers. Figure 24, for example, shows the type of attack produced by the nickel plus 30 percent iron matrix, which was infiltrated at a temperature of 2800° F. It may be seen from the low-magnification photomicrograph (fig. 24) that the fibers have been severely recrystallized. Much of the recrystallized wire, particularly at the edges of the fiber, has been interpenetrated by the nickel-iron alloy, and much of the tungsten has been dissolved. In some cases, the S-816 specimens showed incomplete recrystallization of the fibers, while in others complete recrystallization and dissolution of a considerable portion of the fiber occurred. An external phase exists on the periphery of the fiber, which probably is tungsten-rich. In the case of L-605, which is largely a cobalt-base alloy matrix, again a solution of the fibers and a recrystallization have occurred. In this case, the recrystallization of the centers of the fibers is not as complete as with the S-816 specimens. This is probably the result of a "buffering" type of reaction or a resistance to recrystallization because of the high tungsten content of L-605. On the other hand, the dissolving of a good portion of the fiber into the alloy matrix is evidenced by the shape of the fibers. The peripheral phase is believed to be essentially a high-tungsten intermetallic compound.

Electroplating and "Buffering" Studies

The results obtained from the buffering study give an indication that recrystallization of the tungsten fibers caused by alloying effects may be inhibited by the addition to the alloy of a material that is compatible with the fiber. In this case, niobium and tungsten are not only isomorphous but completely soluble in each other. Thus, it might be possible to select a compatible matrix material and add alloying materials to this that had good corrosion-resisting properties or high-temperature properties. These materials by themselves would tend to recrystallize the fiber, but in the presence of the correct matrix material, they might not recrystallize the fiber.

Bundles of tungsten fibers were electroplated with chromium (the thickness of the plate was about 0.001 in.) and subsequently infiltrated with copper. The bulk of the chromium plate that initially was placed on the fiber remained intact during the infiltration process even though chromium is soluble in copper and vice versa.

One of the interesting aspects of electroplating a fiber with a compatible material such as chromium is the potential of infiltrating other materials around this plated fiber without damaging it by reaction with the alloy infiltrant. In other words, the chromium would be acting as a diffusion barrier by reacting with the matrix rather than allowing the matrix to react with the fiber. Certainly from this experiment, there seems to be a potential for the utilization of such plates to prevent attack on the fibers.

CONCLUSIONS

This investigation was conducted to determine the effect of alloying on the properties and microstructure of tungsten fibers and tungsten-fiber-reinforced composites. Composites were made of tungsten fibers infiltrated with various copper binary alloys (copper was utilized as a "transport medium") containing elements of varying solubility in tungsten.

Metallographic studies were made of a few composites that consist of tungsten fibers infiltrated with high-melting-point alloys. The following conclusions were drawn from this investigation:

1. The mechanical properties of the copper-alloy - tungsten-fiber composites studied were reduced to some degree when alloying with the tungsten fibers occurred. Several of the composite systems tested, however, showed little reduction in tensile strength relative to composites made from the insoluble materials, copper and tungsten, that were used as a base line for comparison. Highly worked tungsten fibers were combined with selected alloying elements without severely reducing their properties.

2. For a given copper-alloy matrix system, it was observed that as the alloying element addition to copper was increased, there was generally an increasing degree of attack on or damage to the properties of the fibers and composites.

3. In general, body-centered cubic alloying elements, which have relatively high solubilities in tungsten and may be expected to exhibit low diffusion rates in tungsten, were less detrimental to the mechanical properties of the composites than face-centered cubic metals. In contrast, face-centered cubic alloying elements, which have low solubilities in tungsten and may be expected to exhibit high diffusion rates in tungsten, were more detrimental to the mechanical properties of the composites.

4. Three types of reactions were observed to have occurred at the metal-matrix - tungsten-fiber interface:

- a. A diffusion-penetration reaction accompanied by a recrystallization of the grains at the periphery of the tungsten fiber
- b. Formation of a two-phase zone
- c. A solid-solution reaction without subsequent recrystallization

Reactions of types b and c did not seriously affect the properties of the fiber or composites studied. The most damaging type of reaction observed was the diffusion-penetration reaction accompanied by recrystallization.

5. Another mechanism by which the composites' strength may be damaged is associated with the ductility behavior of the matrix. It was indicated that some of the copper alloys are brittle because of the intermetallic content of the matrix. In the case of the copper plus 33 percent zirconium alloy binder, cracks form in the matrix and extend into the fibers. In addition, some of the fibers are split in two. This splitting is believed to be the result of a stress-corrosion phenomenon.

6. A correlation between the tensile strength and ductility of the composite was observed. In general, those materials that exhibited the best properties in tension had the best ductilities. The tensile properties and ductility behavior were also correlated with depth-of-penetration measurements. The greater the depth of penetration of the alloying element into the tungsten fibers, the lower the tensile strength and ductility of the composite. An equation relating loss in properties of a composite to depth of penetration of alloying elements into the fibers within the composite was derived by using law-of-mixture equations. By use of the equation, it could be shown that damage to the composite was greater than that predicted by a simple law-of-mixture relation. This fact, along with observed correlations between ductility and tensile strength of the fibers in the composites, suggested that damage was due to a notch-embrittlement effect.

7. It was indicated that the diffusion penetration-recrystallization reaction at the binder-fiber interface caused by several alloy systems studied in this investigation could be prevented by combining this alloying element with one that does not cause this type of reaction and that is compatible with the fiber material.

8. Metallographic examinations of composites of tungsten fibers infiltrated with high-melting-point materials showed that a gross dissolving of the fibers occurred and that in some cases the fibers were either recrystallized or interpenetrated by the infiltrant.

CONCLUDING REMARKS

Relatively few metal-metal systems exist that would permit the creation of fiber-metal composites consisting of mutually insoluble constituents. It is anticipated that most high strength to weight ratio and high-temperature fiber composites ultimately to be produced will utilize high-strength fibers embedded in a highly alloyed matrix. Many high-strength fibers gain a considerable portion of their strength from mechanical deformation processes or thermal treatments and usually contain considerable strain energy. It would not be surprising that, during incorporation of such fibers into a matrix, properties of such high-strength fibers may be considerably reduced by further thermal or mechanical treatments or perhaps by alloying reactions. The findings of this paper are, in some instances, very encouraging in that it has been shown that a very highly stressed fiber will not necessarily be appreciably damaged by alloying reactions. Although all of the alloying additions (of soluble elements) made to the copper-base binder materials lowered the strengths of the composites, some alloying elements did not do more than a superficial damage to the fibers at temperatures at which other elements severely damaged the fibers.

The metallographic studies of the specimens made from the tungsten fibers and the high-melting-point infiltrants helped furnish an understanding of the problems associated with combining high-temperature materials with high-temperature high-strength fibers. Some of the alloys that were infiltrated did severe damage to the fiber by dissolving, cracking, or, in one case, even disintegrating the fibrous product. It is reasonable to expect that some types of coatings or diffusion barriers at the periphery of such fibers could have prevented such catastrophic damage to the fibers. On the other hand, some of the specimens retained a considerable portion of the fiber structure. As long as a composite may be made with a contiguous fiber in the matrix, it is conceivable that it could be restrengthened by such processes as additional working or heat treatments. It should be remembered that the times, temperatures, and manners of infiltration were preselected arbitrarily rather than optimized. It is very possible that, with different infiltration methods and variations in alloying of both fibers and matrices, combinations of high-temperature fibers and higher-melting-point matrices may successfully be made. Certainly, in some cases, combinations of solid-state sintering and mechanical processing techniques might be utilized to prevent severe damage to the fibers during processing. It should be noted that the work of this program was exploratory in nature and may be considered a beginning to the understanding of the effects of alloying elements on the properties of fibers incorporated into high-strength composites.

Lewis Research Center

National Aeronautics and Space Administration

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APPENDIX - DERIVATION OF A MATHEMATICAL EQUATION RELATING THE
DIFFERENCE IN TENSILE STRENGTH BETWEEN A TUNGSTEN-FIBER
COPPER COMPOSITE AND A TUNGSTEN-FIBER COPPER-ALLOY
COMPOSITE WITH A LAW-OF-MIXTURES RELATION ASSUMED

The equation for the predicted tensile strength of a pure copper tungsten-fiber composite, as given in reference 5, is:

$$\left. \begin{aligned} L &= \sigma_{c,Cu} A_c = \sigma_m^* A_m + A_f \sigma_f \\ \sigma_{c,Cu} &= \sigma_m^* A_m + A_f \sigma_f \end{aligned} \right\} \quad (A1)$$

where

L load carried by composite

$\sigma_{c,Cu}$ tensile strength of composite

A_c cross-sectional area of composite (=1)

σ_m^* stress on copper matrix

A_m fraction of cross-sectional area of composite occupied by matrix

σ_f tensile strength of fiber

A_f fraction of cross-sectional area of composite occupied by fiber

Assume that the contribution of the copper alloy to the tensile strength of the composite is approximately equal to that obtained when pure copper is used as a binder. An equation analogous to equation (A1) for a copper-alloy tungsten-fiber composite in which the fibers are partly alloyed would be as follows:

$$\sigma_{c,Cu \text{ alloy}} A_c = \sigma_m^* A_m + \sigma_f (A_f - A_p) + A_p \sigma_p \quad (A2)$$

where

$\sigma_{c,Cu \text{ alloy}}$ tensile strength of alloyed composite

A_p fraction of cross-sectional area of composite occupied by alloyed portion of fiber

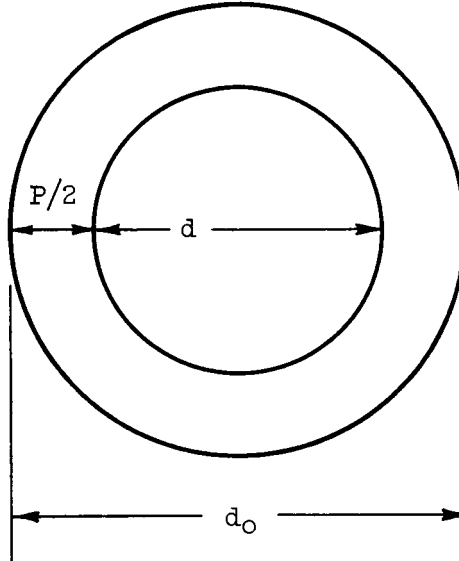
σ_p tensile strength of alloyed zone of fiber

The difference in tensile strength of a copper-alloy tungsten-fiber composite relative to a copper tungsten-fiber composite with the same initial fiber content assumed is given by subtracting equation (A2) from (A1). The following

equation is obtained:

$$\sigma_{c,Cu} - \sigma_{c,Cu \text{ alloy}} = A_p(\sigma_f - \sigma_p) \quad (A3)$$

Express equation (A3) in terms of the depth of the alloyed fiber zone, which is designated as $P/2$. The original diameter of the fiber is given as d_o , and the unalloyed zone as d (see sketch).



The symbol A_p may be expressed in terms of P and A_f (the original fiber content) as follows:

$$A_p = \left(\frac{d_o^2 - d^2}{d_o^2} \right) A_f$$

Since $d = d_o - P$,

$$A_p = \left[\frac{d_o^2 - (d_o^2 + P^2 - 2d_o P)}{d_o^2} \right] A_f$$

$$A_p = \left(\frac{2d_o P - P^2}{d_o^2} \right) A_f$$

Substituting in equation (A3) yields

$$\sigma_{c,Cu} - \sigma_{c,Cu \text{ alloy}} = (\sigma_f - \sigma_p) \left[\frac{A_f(2d_o P - P^2)}{d_o^2} \right]$$

Let

$$\Delta\sigma_c = \sigma_{c,Cu} - \sigma_{c,Cu \text{ alloy}}$$

and

$$K = \frac{(\sigma_f - \sigma_p)}{d_o^2} \quad (A4)$$

Then

$$\Delta\sigma_c = KA_f(2Pd_o - P^2)$$

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TABLE I. - ROOM TEMPERATURE TENSILE PROPERTIES OF COPPER-ALLOY - TUNGSTEN-FIBER COMPOSITES

Binder material	Maximum solubility of alloying element in tungsten	Weight percent of alloying element	Atomic percent of alloying element	Specimen	Volume percent fiber	Tensile strength, psi	Reduction in area, percent	Type of fracture
Pure copper (ref. 5)	Insoluble in tungsten	0	0	--	65	225,700	----	Ductile
				--	70.2	238,000	----	Ductile
				--	75.4	249,800	----	Ductile
Copper-nickel	0.3	5	5.4	1	79	246,600	34	Ductile
				2	78.4	250,000	37	Ductile
				3	76	218,900	32	Ductile
Copper-cobalt	0.3	10	10.9	4	74.1	131,700	Nil	Brittle
				5	75.5	108,800	Nil	Brittle
				6	79.5	51,300	Nil	Brittle
Copper-cobalt	0.3	1	1.1	7	77.3	219,400	----	Semiductile
		5	5.4	8	76	213,200	1.5	Semiductile
				9	74.8	229,300	2.3	Ductile
Copper-aluminum	2.6			10	74.7	147,200	----	Brittle
				11	74.9	172,100	----	Brittle
		5	11.3	12	63.4	98,900	Nil	Brittle
Copper-titanium	8			13	72.4	153,800	Nil	Semiductile
				14	76.1	154,500	Nil	Semiductile
		10	20.8	15	76.7	138,500	----	Brittle
Copper-zirconium	3	10	12.8	16	78.2	223,700	----	Semiductile
				17	71.7	220,100	10	Semiductile
		25	30.7	18	76.3	186,700	----	Brittle
Copper-chromium	Complete solid solubility (Miscibility gap)	10	7.2	19	72.8	216,000	Nil	Brittle
				20	78.5	255,300	↓	Ductile
				21	75.6	226,900		Semiductile
Copper-niobium	Complete solid solubility			22	64.7	172,600		Brittle
				23	64.3	195,700		Semiductile
		33	25.5	24	75.9	106,700	Nil	Brittle
Copper-chromium	Complete solid solubility (Miscibility gap)	1	1.2	25	78.7	223,500	7.4	Semiductile
				26	77.5	228,600	25.8	Ductile
				27	77.2	225,900	7.5	Semiductile
Copper-niobium	Complete solid solubility	2	2.4	28	76.4	241,700	16.4	Ductile
		1	0.6	29	75.4	237,100	20.6	Ductile
				30	75.1	223,100	24.7	Ductile

TABLE II. - COMPOSITION OF COMMERCIAL ALLOYS

Alloy	Nominal composition									
	Co	Cr	Ni	W	Nb	Mo	Fe	Si	Mn	C
S-816	45	20	20	4	4	3	2	0.3	0.6	0.4
L-605	Bal.	20	10	15	--	--	3	1	1.5	0.1

TABLE III. - METAL-FIBER INTERFACE REACTION ZONE MEASUREMENTS

Binder material	Type of reaction	Measurements of reactions (average values)			
		Recrystallization- penetration zone, in.	External plate, in.	Two-phase zone, in.	Penetration solid- solution zone, in.
Cu- 5% Al	Diffusion penetration- recrystallization	0.0009	-----	-----	-----
Cu-10% Al	Diffusion penetration- recrystallization	.00104	0.000154	-----	-----
Cu- 1% Co	Diffusion penetration- recrystallization	.000362	-----	-----	-----
Cu- 5% Co	Diffusion penetration- recrystallization	.000580	.000138	-----	-----
Cu- 1% Cr	Diffusion penetration- recrystallization	-----	-----	-----	0.00011
Cu- 2% Cr	Penetration-solid solution	-----	-----	-----	.00011
Cu- 1% Nb	Penetration-solid solution	-----	-----	-----	a .000052
Cu- 5% Ni	Diffusion penetration	-----	-----	-----	a .000052
Cu-10% Ni	Diffusion penetration- recrystallization	.000666	.000238	-----	-----
Cu-10% Ti	Two-phase zone	-----	-----	a 0.000052	-----
Cu-25% Ti	Two-phase zone	-----	-----	.000880	-----
Cu-10% Zr	Two-phase zone	-----	-----	(b)	-----
Cu-33% Zr	Two-phase zone	-----	-----	a .000052	-----
Ni-30% Fe	Fiber dissolution- recrystallization	.005	-----	-----	-----
Nb-48% Ni	Two-phase zone	-----	-----	.000335	-----
L-605	Fiber dissolution- recrystallization	c .0012	.000666	-----	-----
S-816	Fiber dissolution- recrystallization	c .0025	.00035	-----	-----
Chromium-plated wires	Plating plus solid- solution zone	-----	.001	-----	.0008

aEstimated.

bNot measurable.

cPortions of the fibers were dissolved. The average wire diameters after infiltration were: L-605, 0.0045 in.; S-816, 0.0041 in.

TABLE IV. - AVERAGE DECREASE AND PERCENT REDUCTION IN TENSILE STRENGTH OF
TUNGSTEN-FIBER-REINFORCED - COPPER-ALLOY COMPOSITES RELATIVE TO
TUNGSTEN-FIBER-REINFORCED - COPPER COMPOSITES

Binder material	Average decrease in tensile strength, psi	Average percent reduction in tensile strength
Cu-10% Ni	160,000	62
^a Cu-33% Zr	143,000	57
Cu-10% Al	117,000	46
Cu- 5% Al	100,000	43
^a Cu-25% Ti	68,000	27
Cu- 5% Co	62,000	24
Cu-10% Ti	37,000	14
Cu- 1% Cr	30,000	12
Cu-10% Zr	25,000	10
Cu- 1% Nb	20,000	8
Cu- 5% Ni	17,500	7

^aAlthough these alloys produced two-phase microstructures at the periphery of the fibers (within the fibers), the reduced tensile properties are believed to be the result of the embrittlement of the matrix by the large quantities of the alloying elements added to copper.

TABLE V. - PHASE EQUILIBRIUM RELATIONS

[Refs. 18 and 20.]

Element	Melting point		Crystal structure	Maximum solubility of element in tungsten, weight percent	Maximum solubility of tungsten in element, weight percent	Eutectic formation, °C	Probable intermetallic
	°F	°C					
Al	1220	660	Face-centered cubic	2.6	1.5	----	WAl ₇ , WAl ₁₂ , W ₂ Al, WAl ₃ , WAl ₄ , WAl ₅
Co	2723	1495	Close-packed hexagonal (below 788° F) face-centered cubic (above 788° F)	0.3	45	1480	WCo ₃ , W ₆ Co ₇
Cr	3430	1890	Close-packed hexagonal (below 82° F) body-centered cubic (above 82° F)	Complete solid solubility (Miscibility gap)		----	-----
Nb	4380	2415	Body-centered cubic	Complete solid solubility		----	-----
Ni	2651	1455	Face-centered cubic	0.3	45	1500	WNi ₄
Ti	3300	1820	Close-packed hexagonal (below 1616° F) body-centered cubic (above 1616° F)	8	50	1880	-----
Zr	3200	1750	Close-packed hexagonal (below 862° F) body-centered cubic (above 862° F)	3	8	1660	W ₂ Zr

TABLE VI. - PENETRATION-RECRYSTALLIZATION ZONES COMPARED WITH TENSILE STRENGTH OF TUNGSTEN-FIBER-REINFORCED - COPPER PLUS 5 PERCENT COBALT COMPOSITES

Specimen	Penetration-recrystallization zone, in.	Tensile strength, psi	Volume percent fibers
9	0.000381	229,300	74.77
8	.000425	213,200	75.96
11	.000675	172,100	74.88
10	.000840	147,200	74.73

TABLE VII. - COMPARISON OF DUCTILITY AND DEPTH OF PENETRATION WITH K

Alloy	Specimen	Type of fracture	Change in tensile strength of composite, $\Delta\sigma_c$, psi	Cross-sectional area of composite occupied by fiber, A_f , percent	Twice the depth of penetration, P, in.	$K = \frac{\sigma_f - \sigma_p}{d_o^2}$, lb/in. ⁴
Cu- 5% Ni	1	Ductile	18,400	79	0.000106	223x10 ⁻⁸
	2	Ductile	10,000	78.4	.000106	122
	3	Ductile	36,000	76	.000106	452
Cu-10% Ni	4	Brittle	115,800	74.1	0.00087	195x10 ⁻⁸
	5	Brittle	143,000	75.5	.00158	143
	6	Brittle	214,000	79.5	.00155	206
Cu- 5% Co	8	Semiductile	42,000	76	0.00085	71x10 ⁻⁸
	9	Ductile	21,000	74.8	.00076	40
	10	Brittle	103,000	74.7	.00168	99
	11	Brittle	78,000	74.9	.00135	89
Cu-10% Ti	16	Semiductile	38,300	78.2	a0.000104	476x10 ⁻⁸
	17	Semiductile	38,000	71.7	.000104	515
Cu-10% Zr	19	Brittle	26,000	72.8	a0.000104	347x10 ⁻⁸
	20	Ductile	5,000	78.5	↓	64
	21	Semiductile	23,000	75.6		296
	22	Brittle	43,400	64.8		645
	23	Semiductile	17,300	64.3		262
Cu- 1% Nb	29	Ductile	15,000	75.4	a0.000104	193x10 ⁻⁸
	30	Ductile	28,000	75.1	.000104	362

^aEstimated value

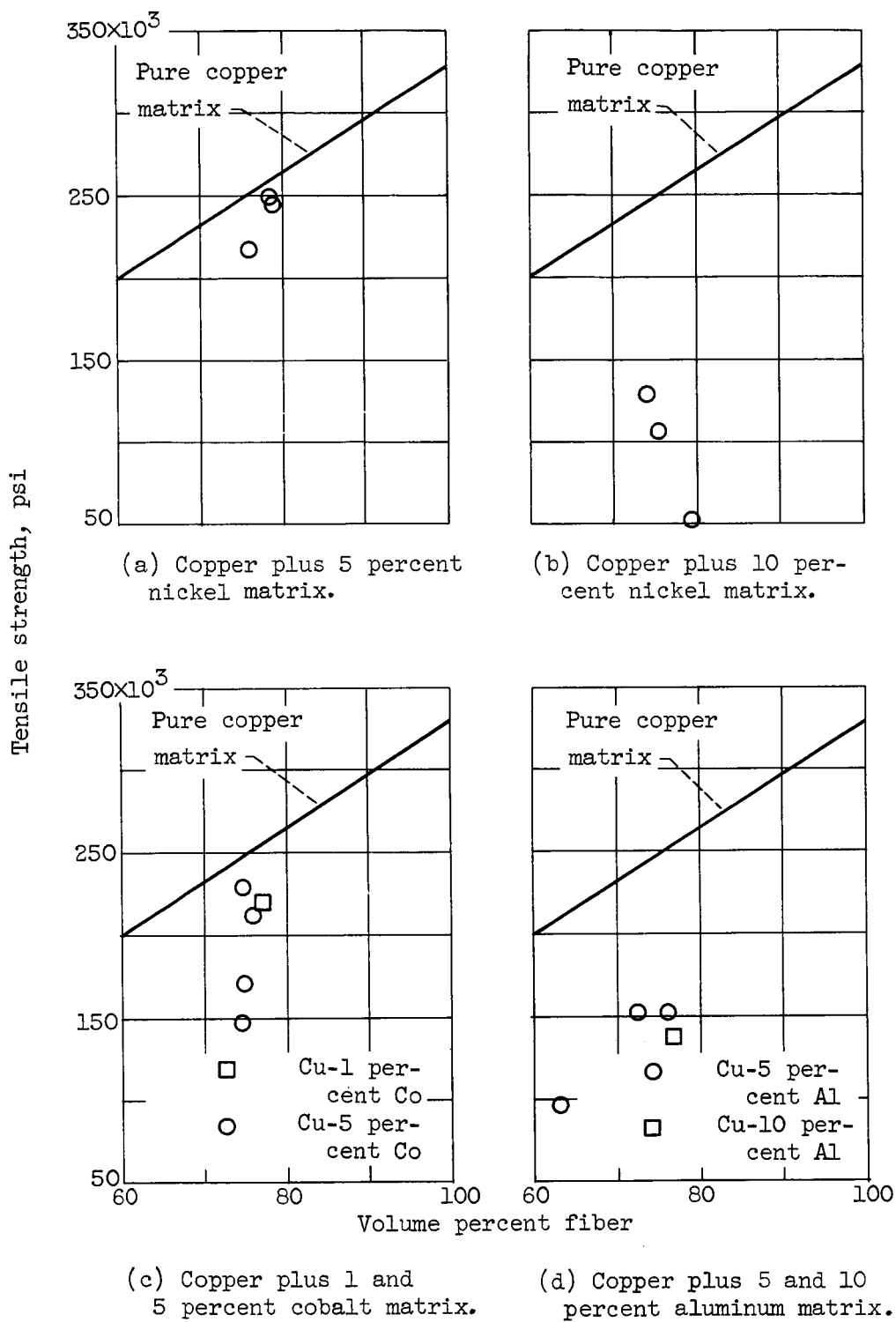


Figure 1. - Strength-composition diagrams comparing copper - tungsten-fiber composites to copper-alloy - tungsten-fiber composites.

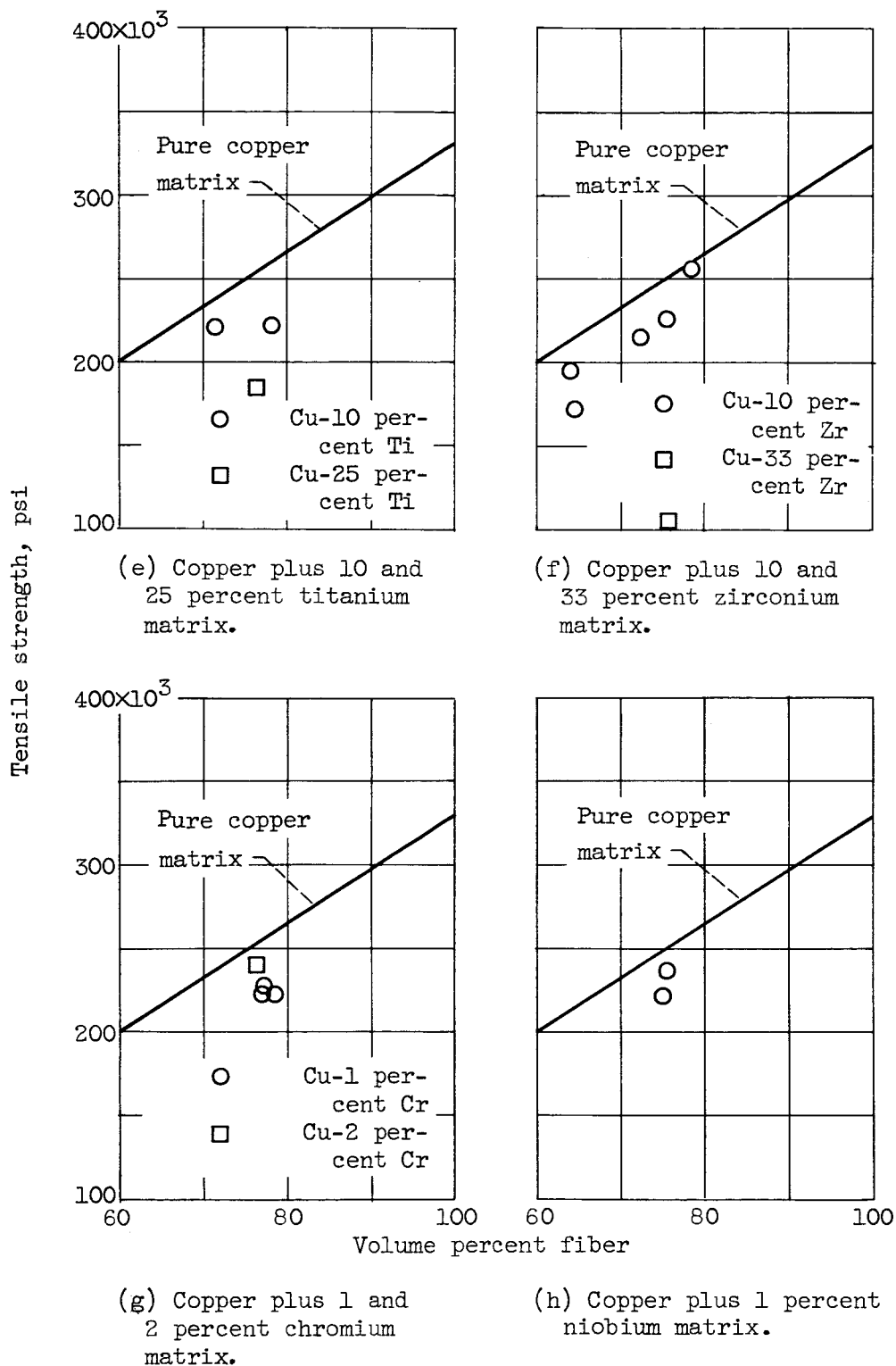
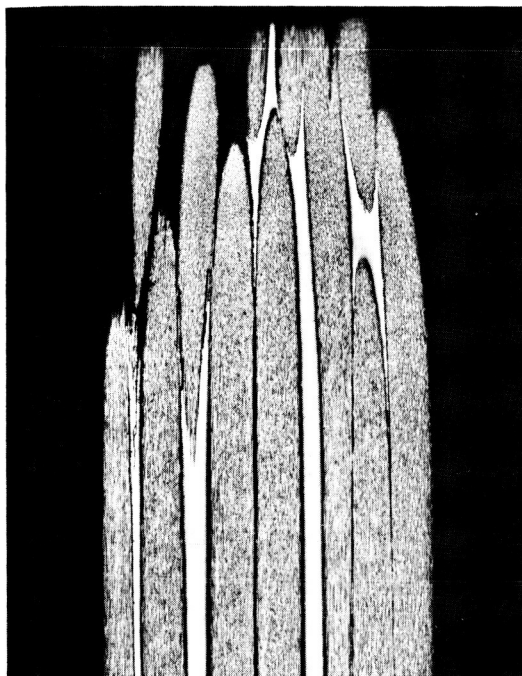
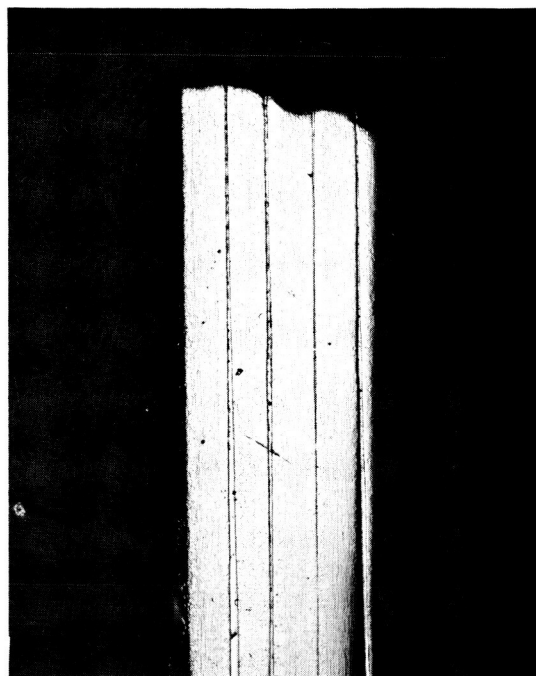


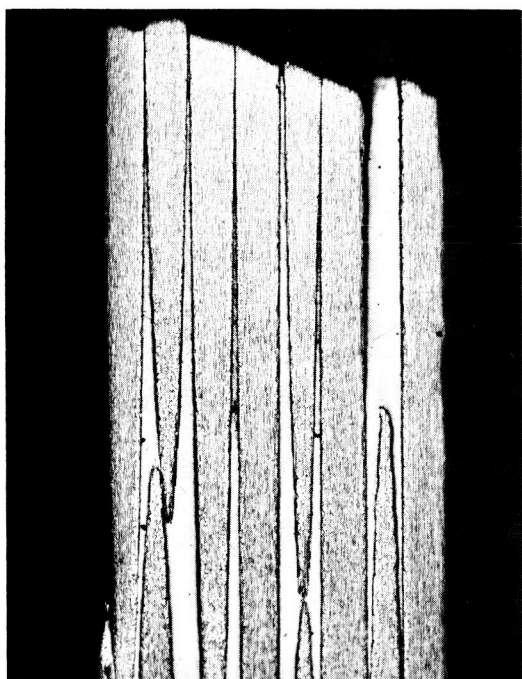
Figure 1. - Concluded. Strength-composition diagrams comparing copper - tungsten-fiber composites to copper-alloy - tungsten-fiber composites.



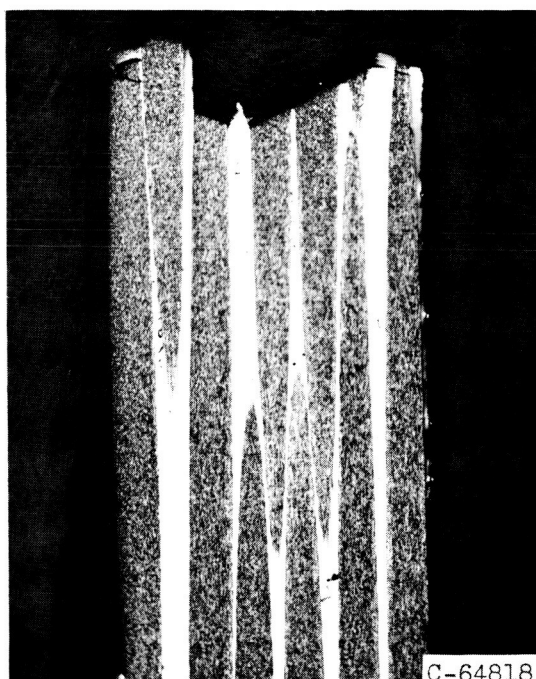
(a) Copper plus 5 percent nickel.



(b) Copper plus 10 percent nickel.

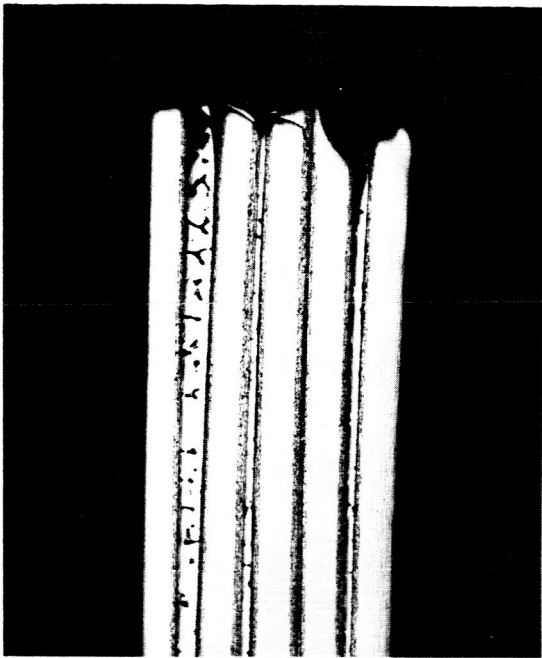


(c) Copper plus 5 percent cobalt.

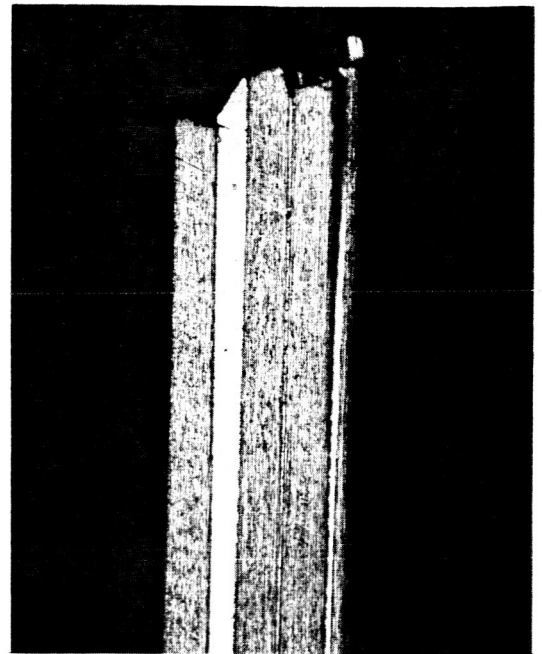


(d) Copper plus 5 percent aluminium.

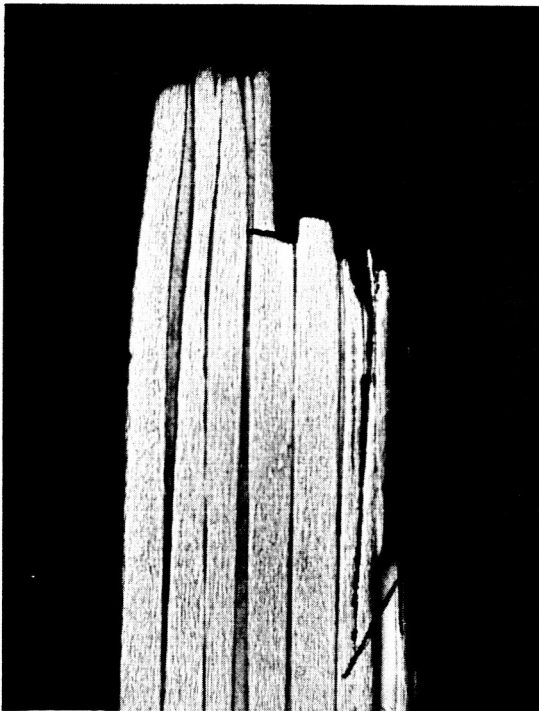
Figure 2. - Typical fracture edges of copper-alloy - tungsten-fiber composites.



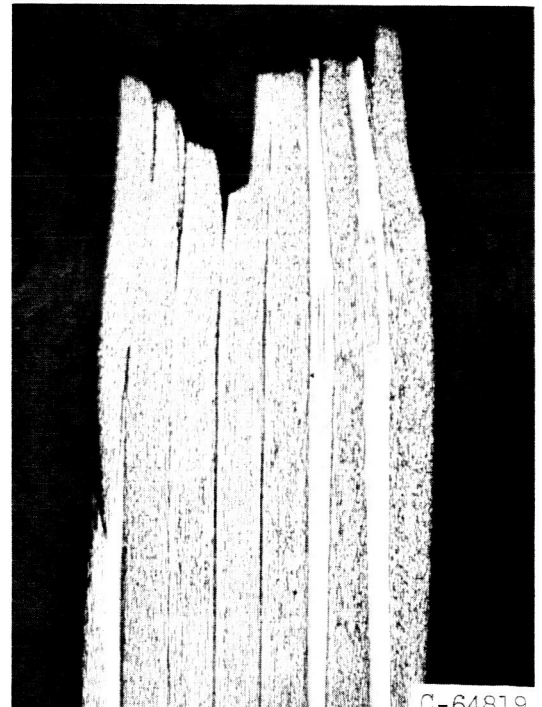
(e) Copper plus 10 percent titanium.



(f) Copper plus 10 percent zirconium.

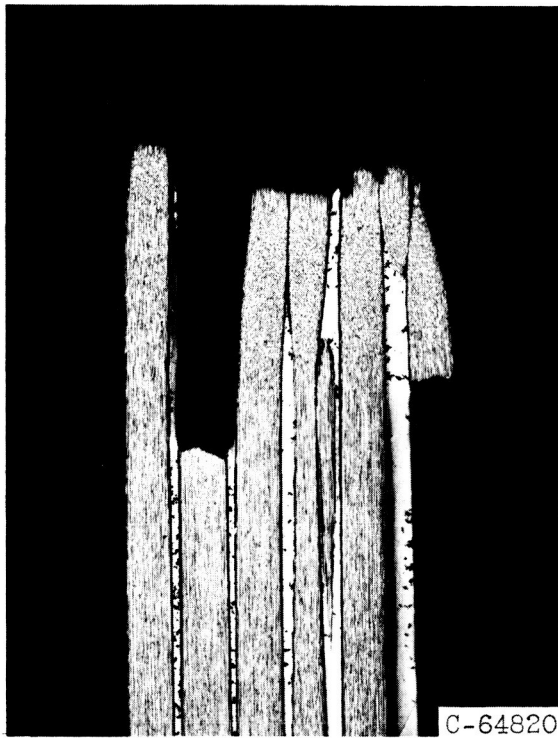


(g) Copper plus 1 percent chromium.



(h) Copper plus 2 percent chromium.

Figure 2. - Continued. Typical fracture edges of copper-alloy - tungsten-fiber composites.



(i) Copper plus 1 percent niobium.

Figure 2. - Concluded. Typical fracture edges of copper-alloy - tungsten-fiber composites.

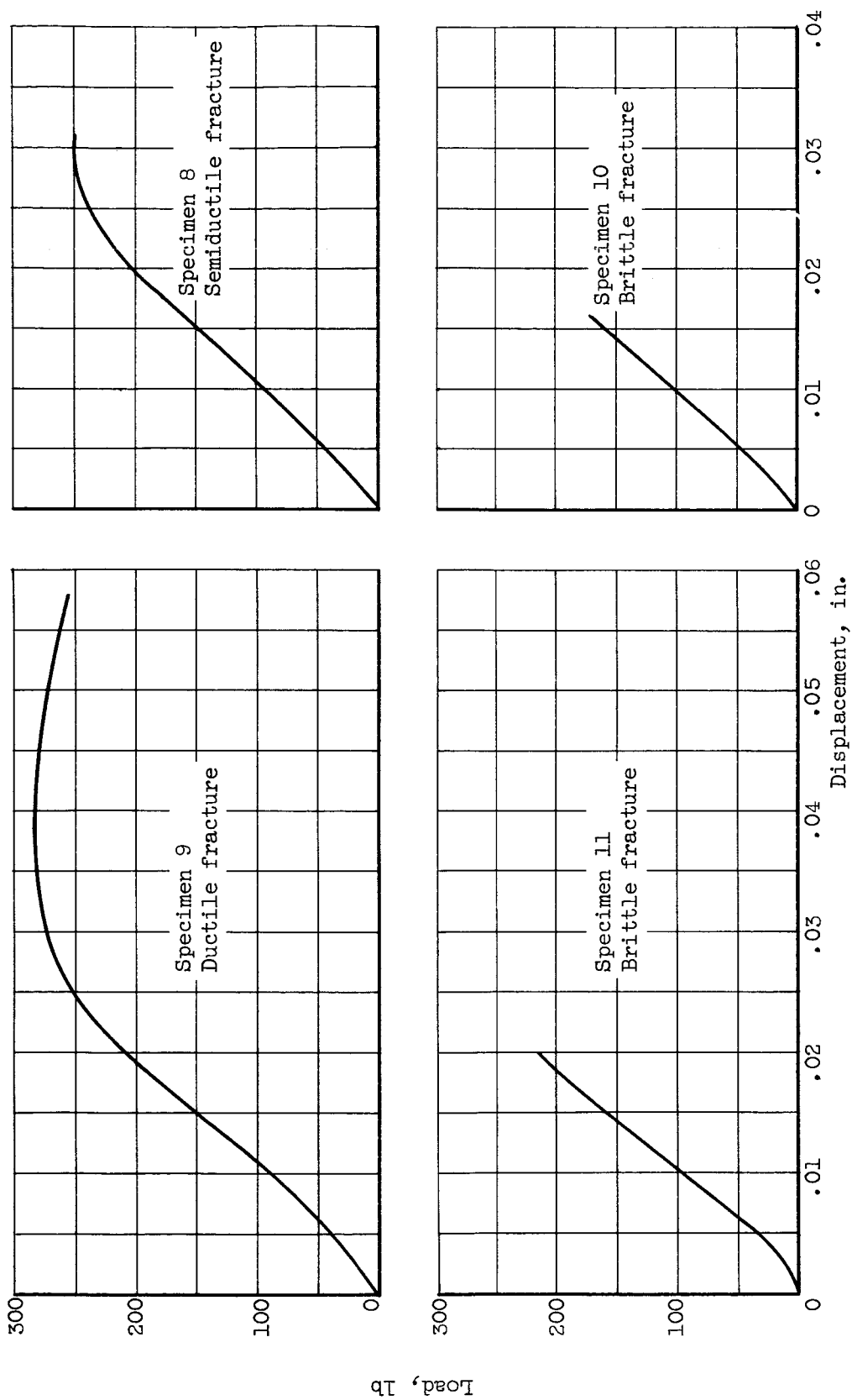
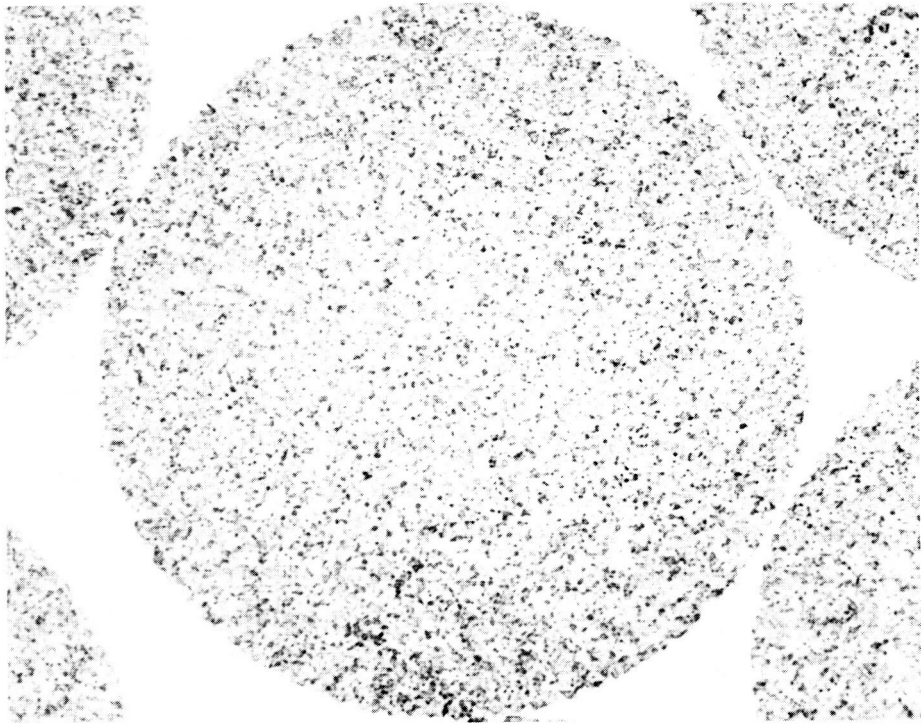
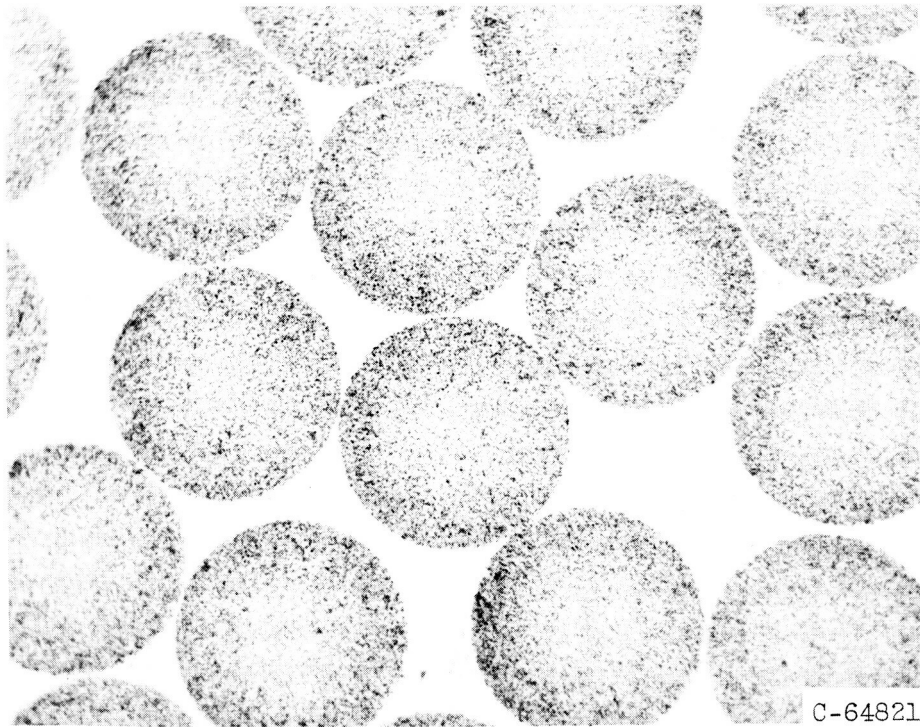


Figure 3. - Load as a function of displacement curves for tungsten-fiber-reinforced - copper plus 5 percent cobalt composites.



x750



x250

Figure 4. - Pure copper matrix.

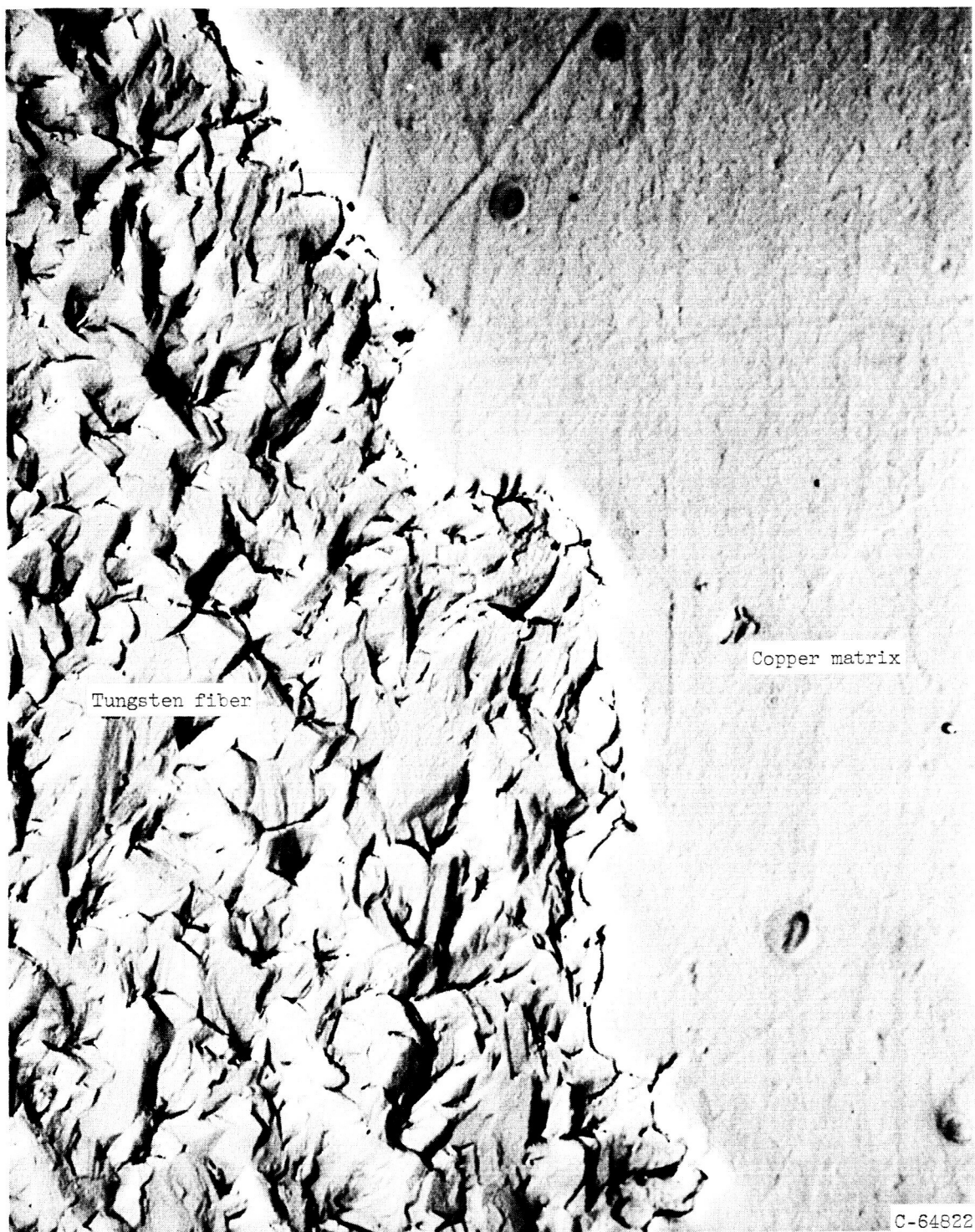
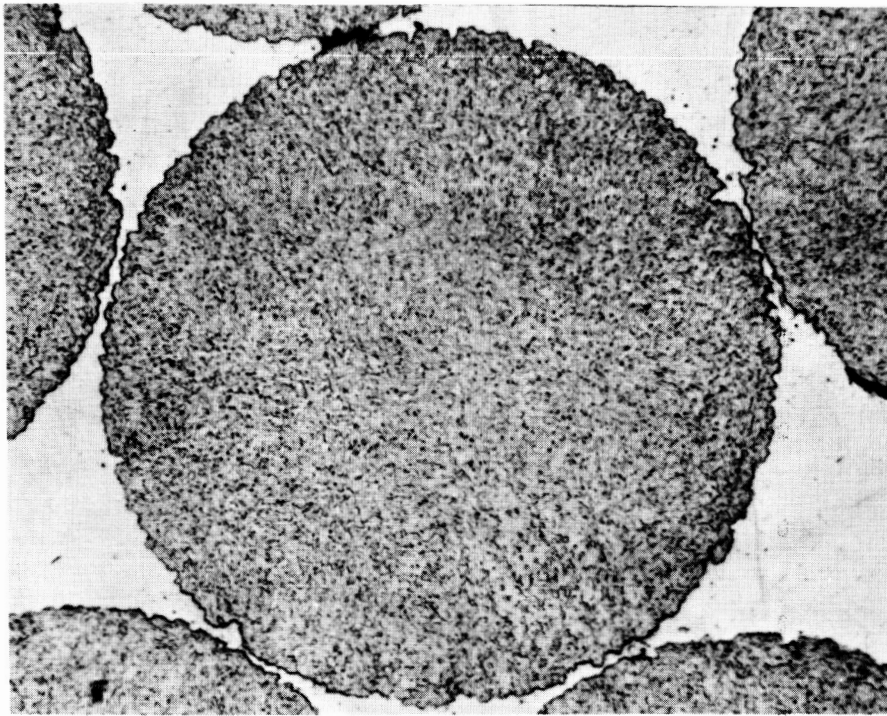
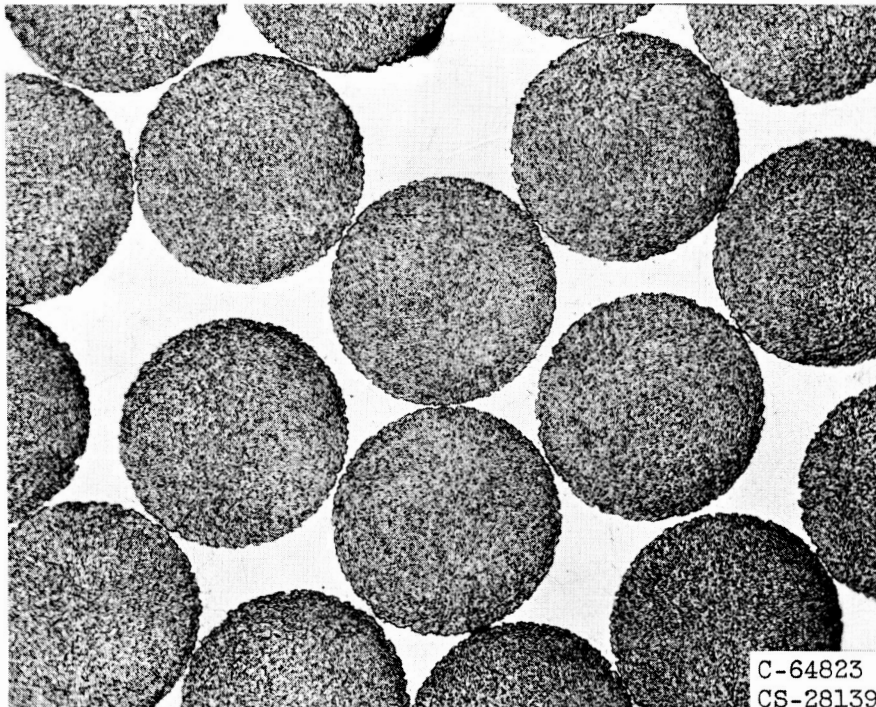


Figure 5. - Electron photomicrograph of copper-matrix - tungsten-fiber composite.

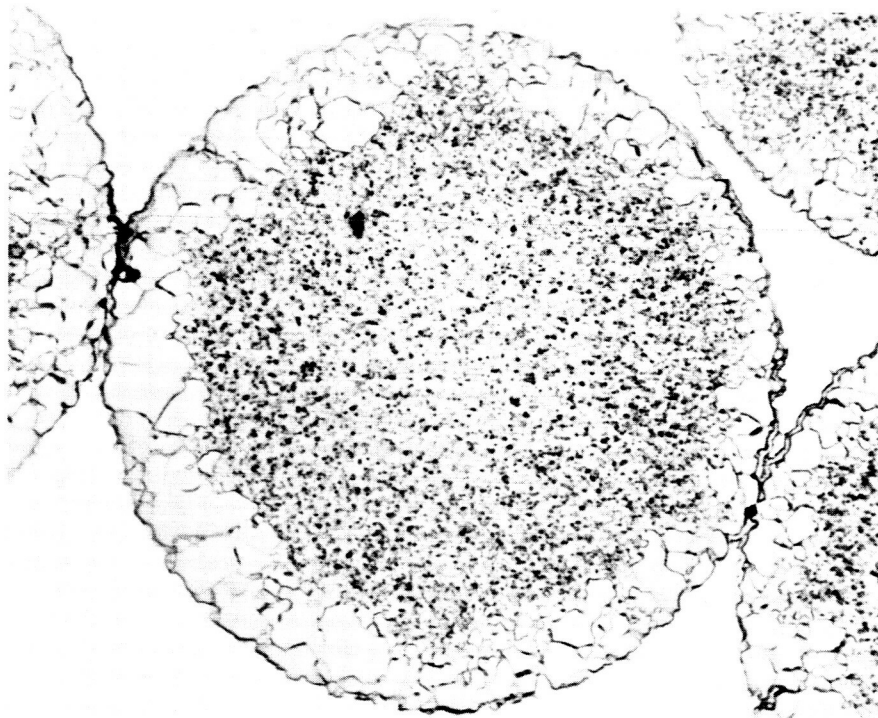


x750

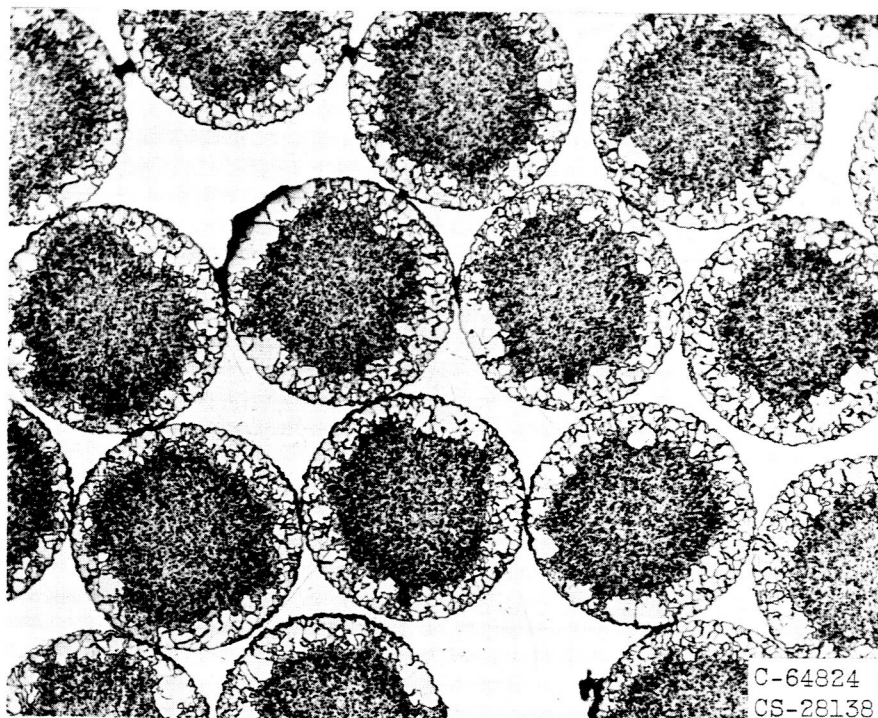


x250

Figure 6. - Copper plus 5 percent nickel matrix.



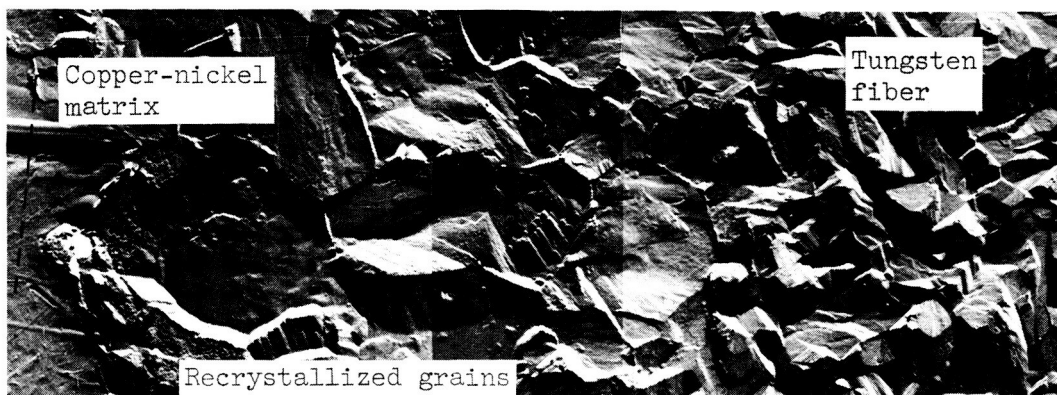
x750



C-64824
CS-28138

x250

Figure 7. - Copper plus 10 percent nickel matrix.



x26,000



x26,000



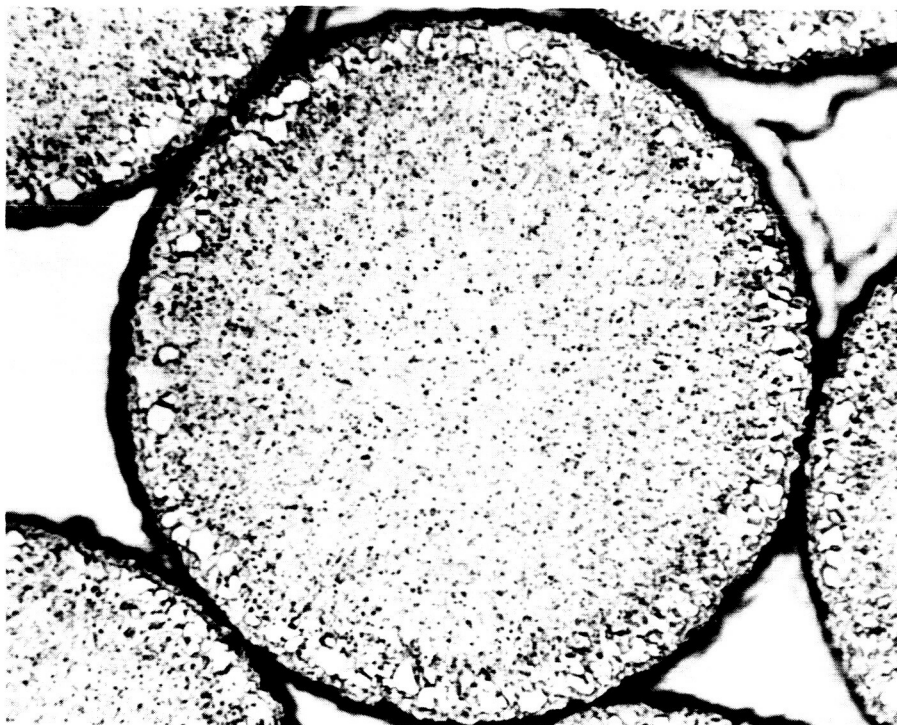
x26,000



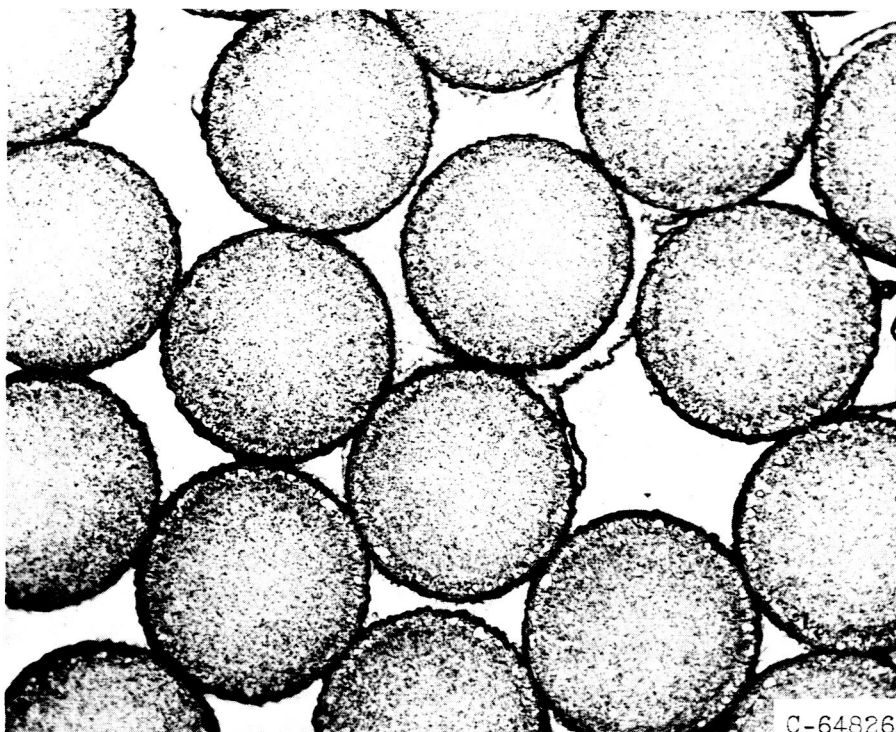
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x13,000

Figure 8. - Copper plus 10 percent nickel matrix.



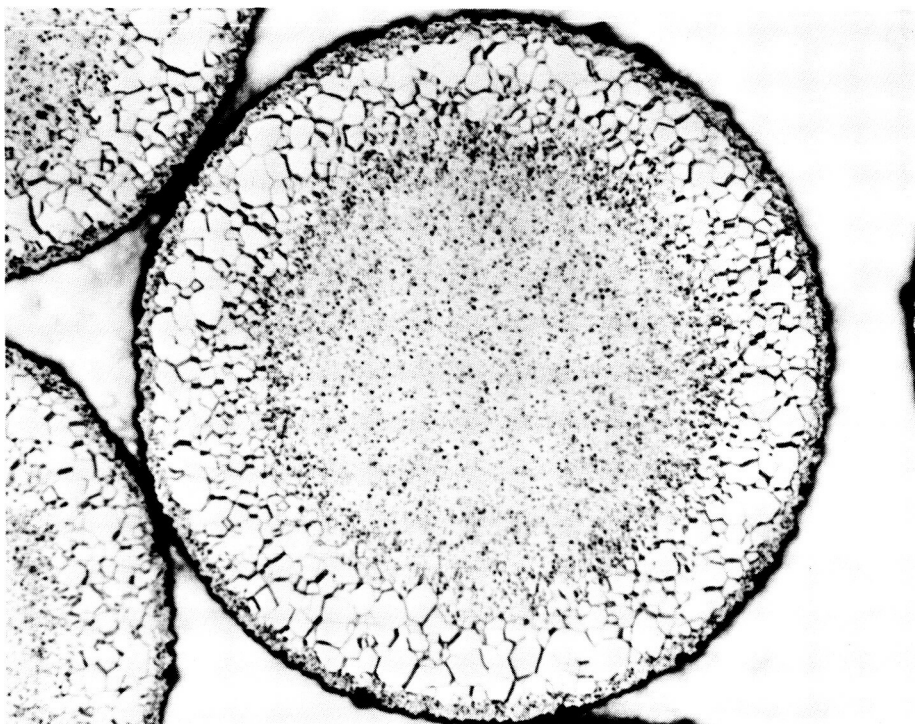
x750



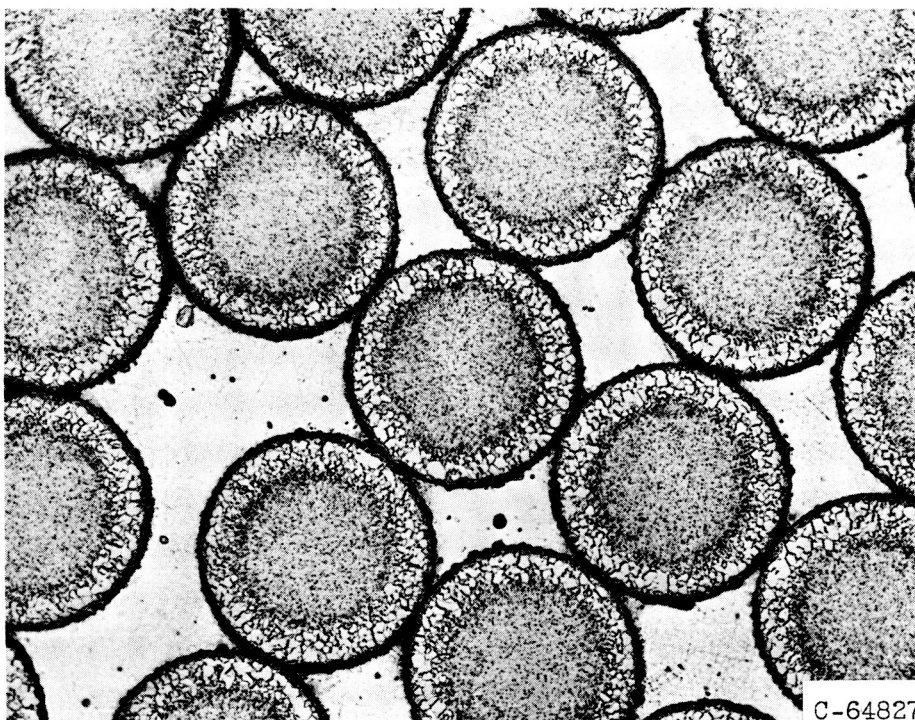
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x250

Figure 9. - Copper plus 1 percent cobalt matrix.



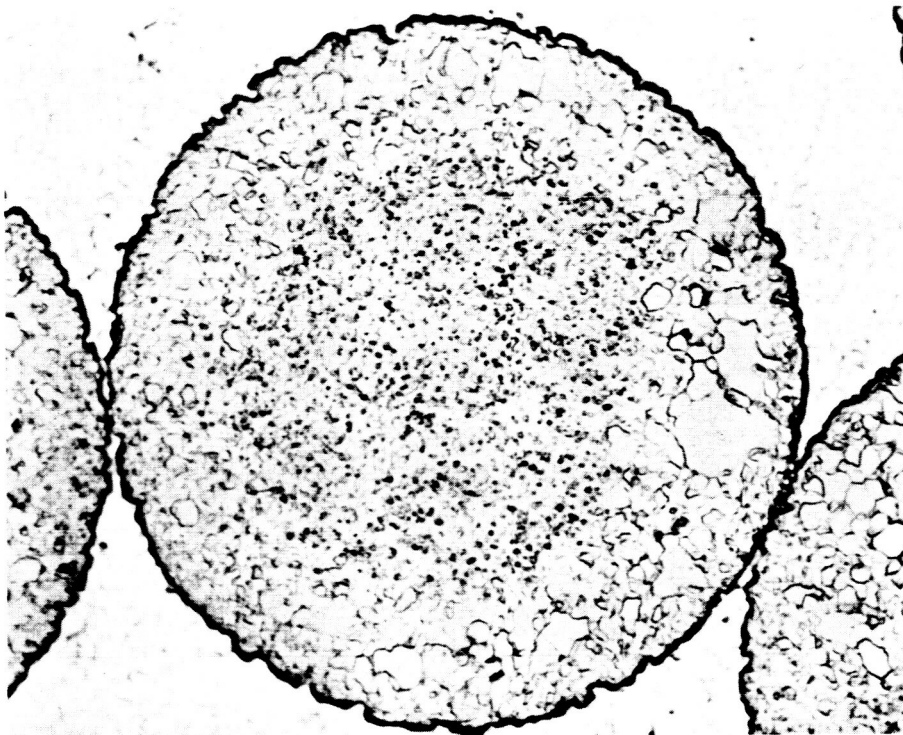
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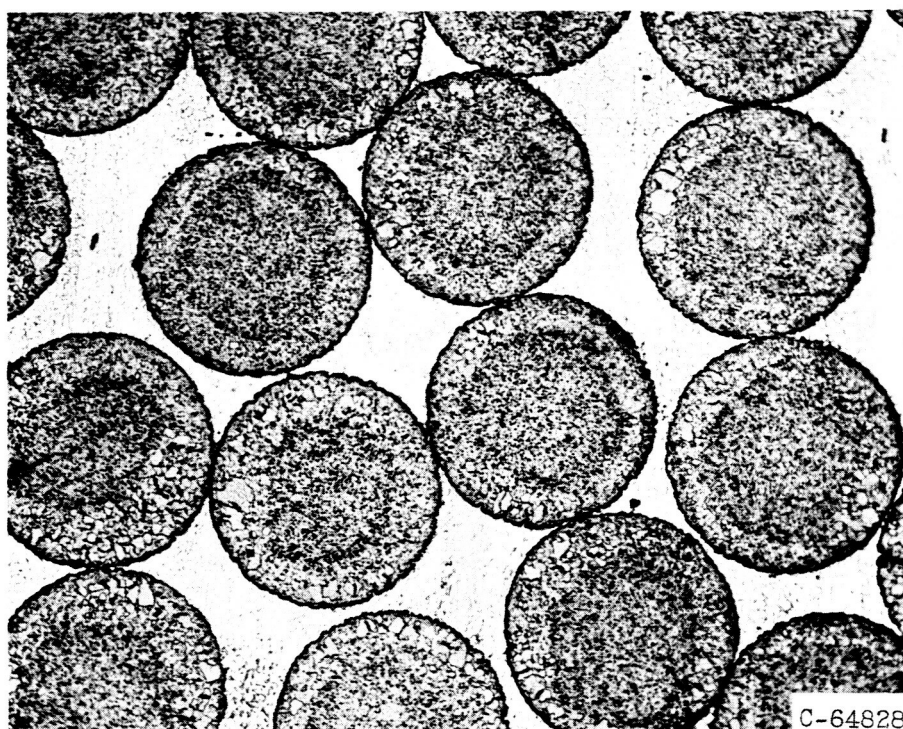
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x250

Figure 10. - Copper plus 5 percent cobalt matrix.

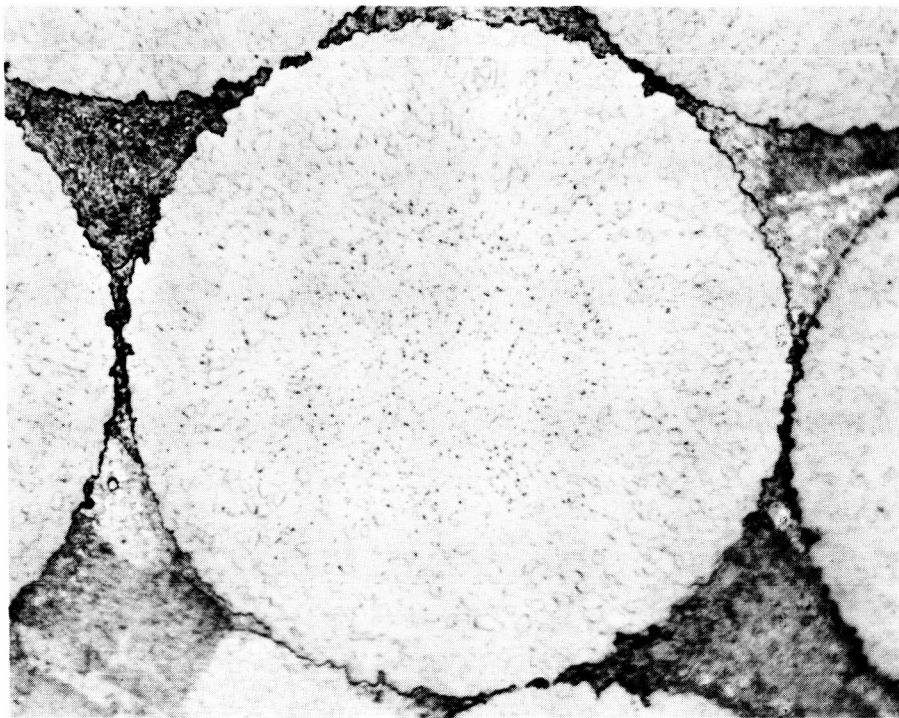


X750

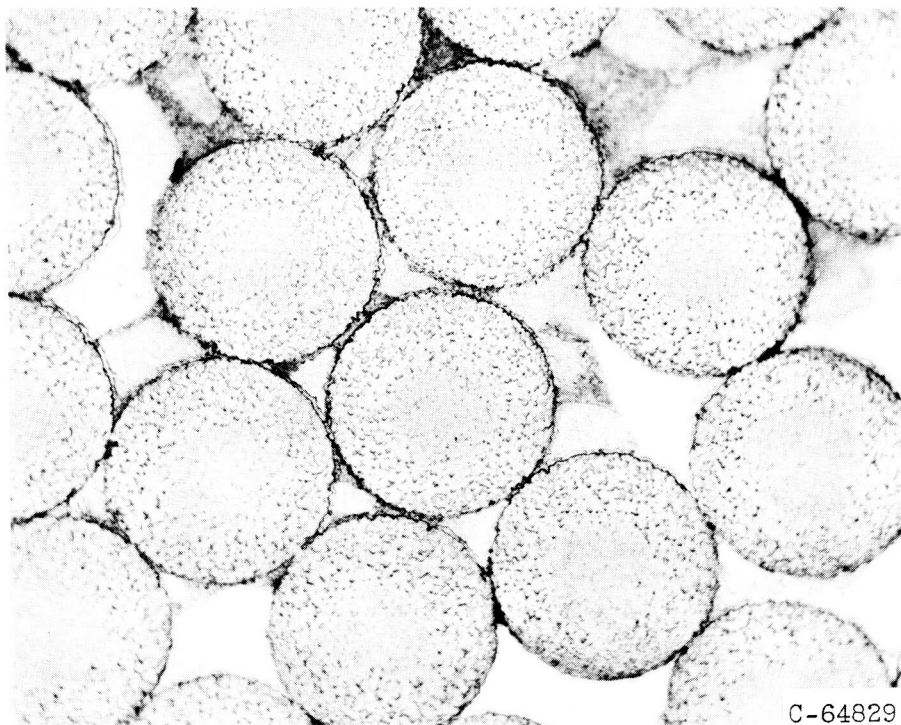


X250

Figure 11. - Copper plus 5 percent aluminium matrix.



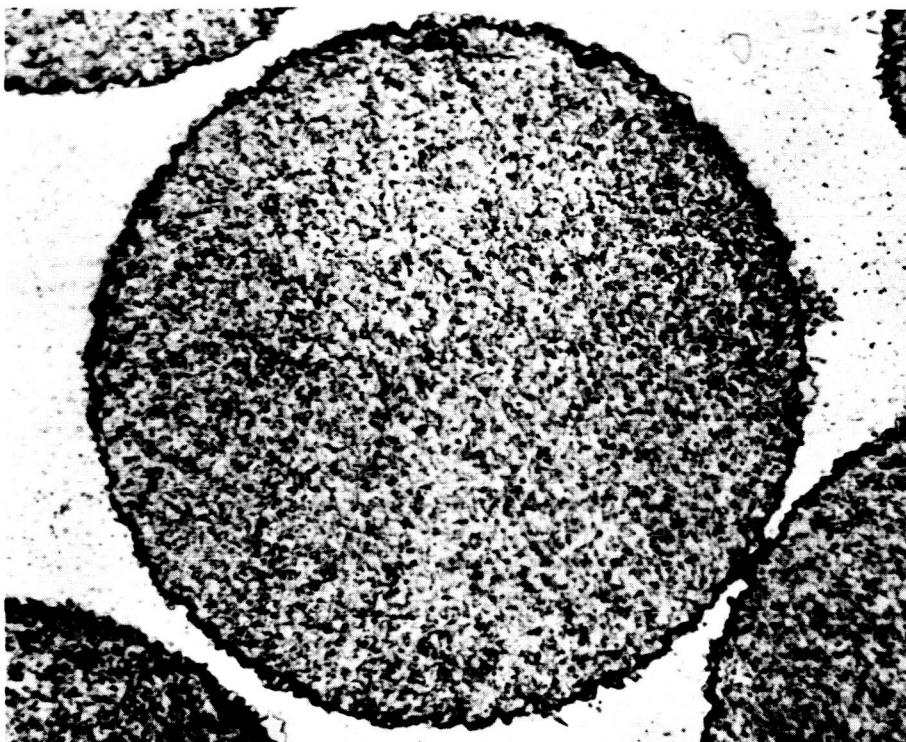
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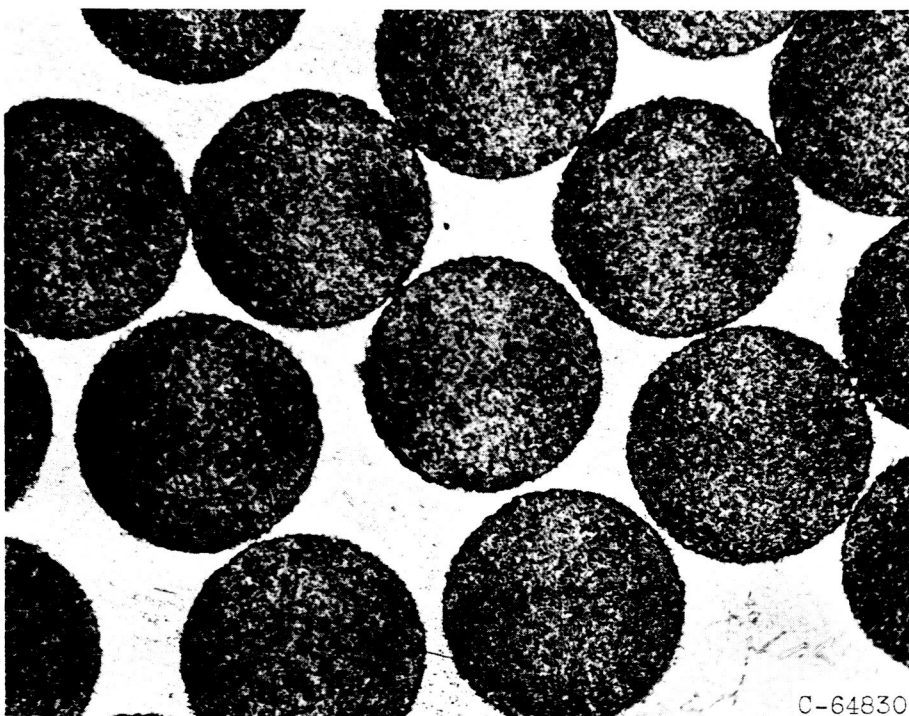
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x250

Figure 12. - Copper plus 10 percent aluminium matrix.



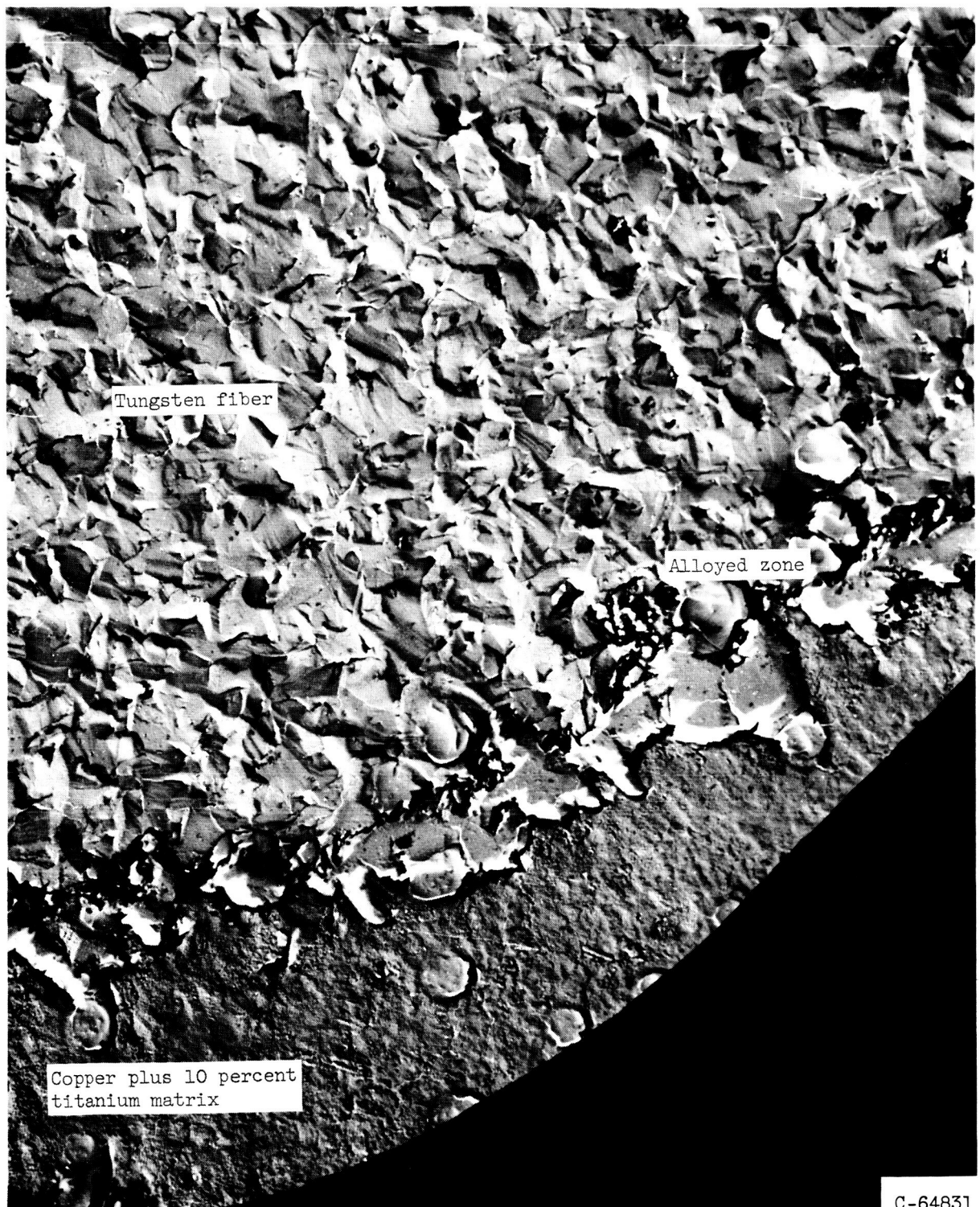
X750



C-64830

X250

Figure 13. - Copper plus 10 percent titanium matrix.



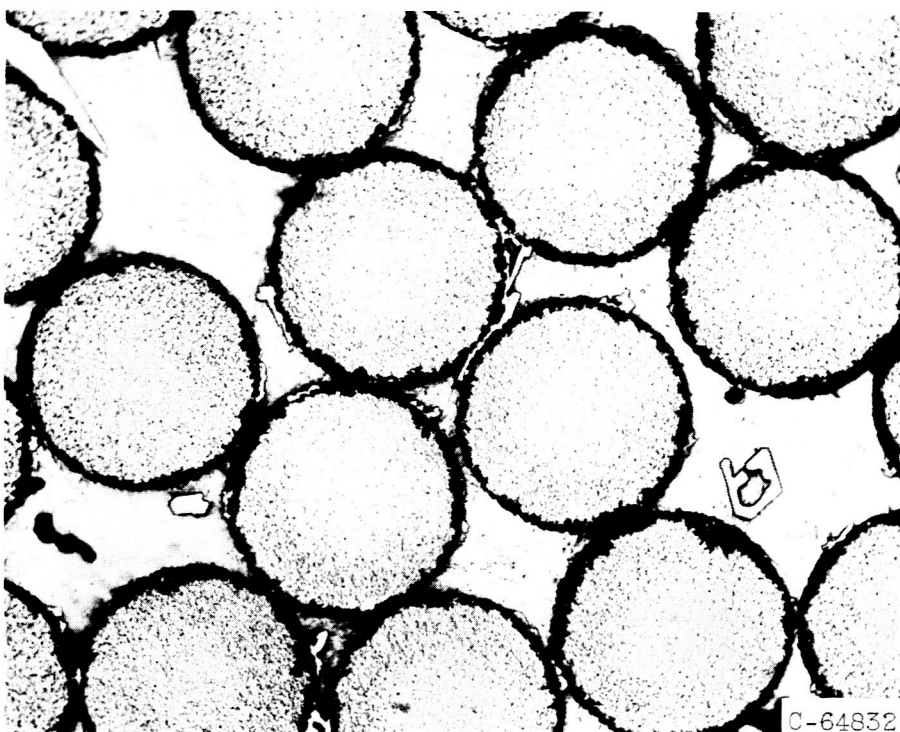
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×12,000

Figure 14. - Electron photomicrograph of copper plus 10 percent titanium matrix tungsten-fiber composite.

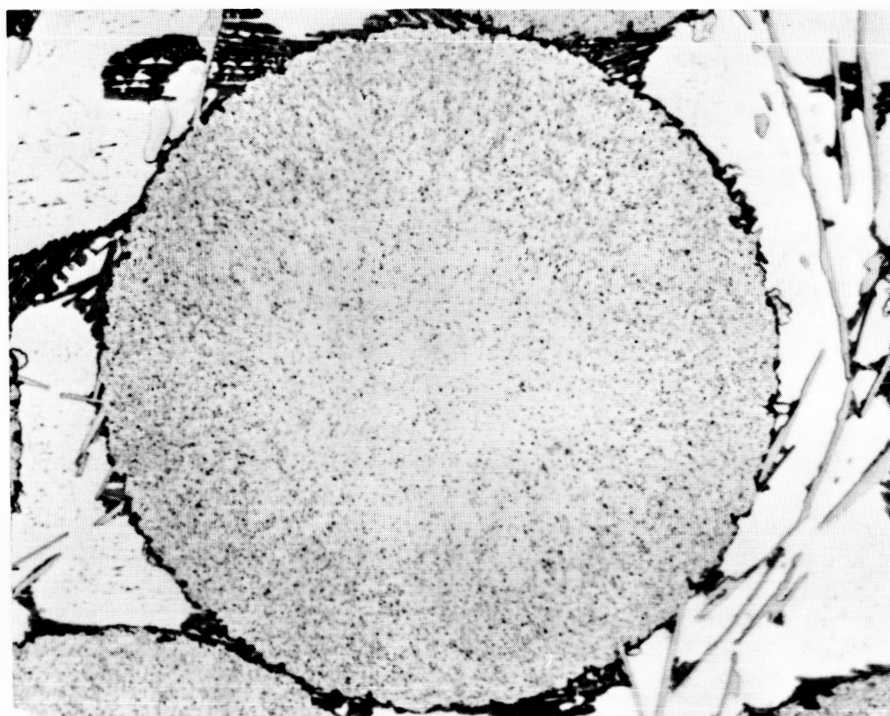


x750

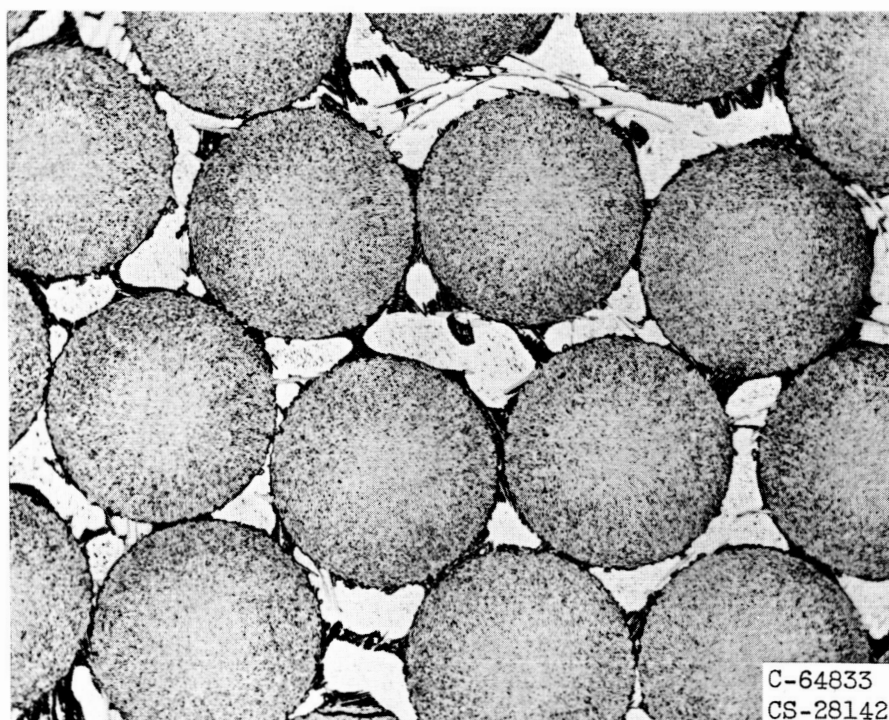


x250

Figure 15. - Copper plus 25 percent titanium matrix.



x750



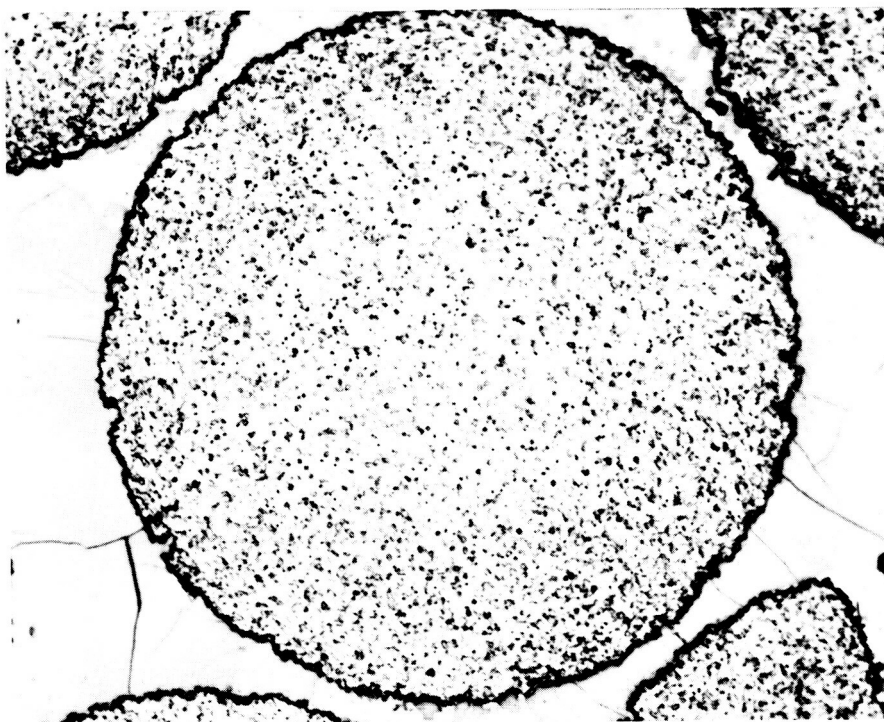
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Figure 16. - Copper plus 10 percent zirconium matrix.

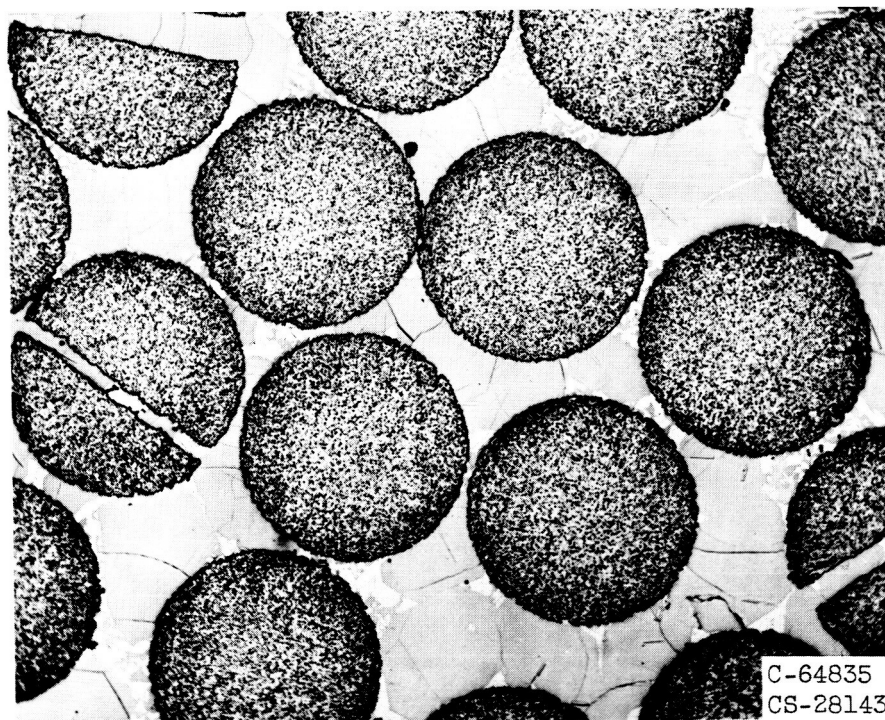


x12,000

Figure 17. - Electron photomicrograph of copper plus 10 percent zirconium tungsten-fiber composite.

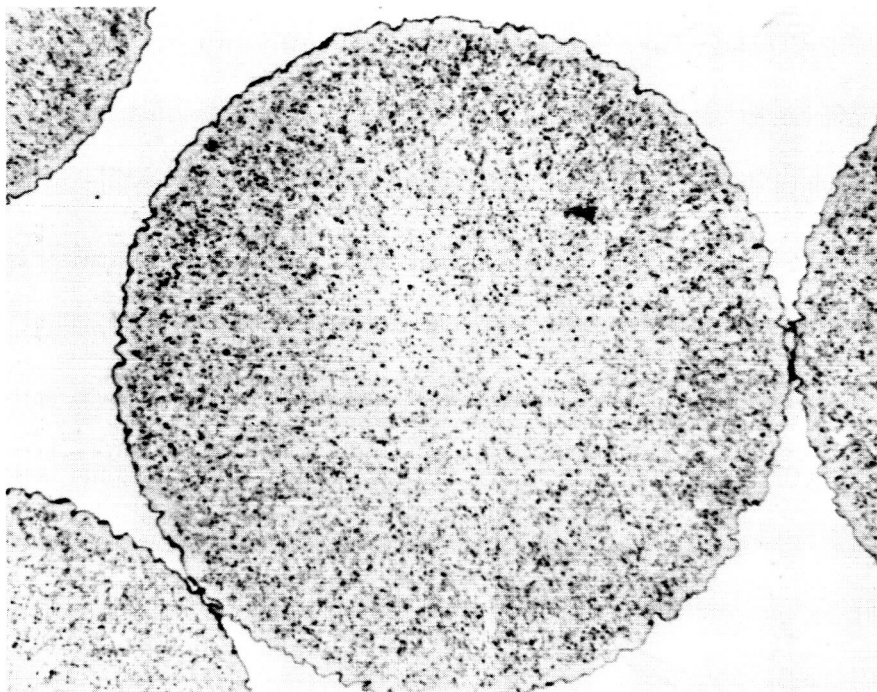


x750

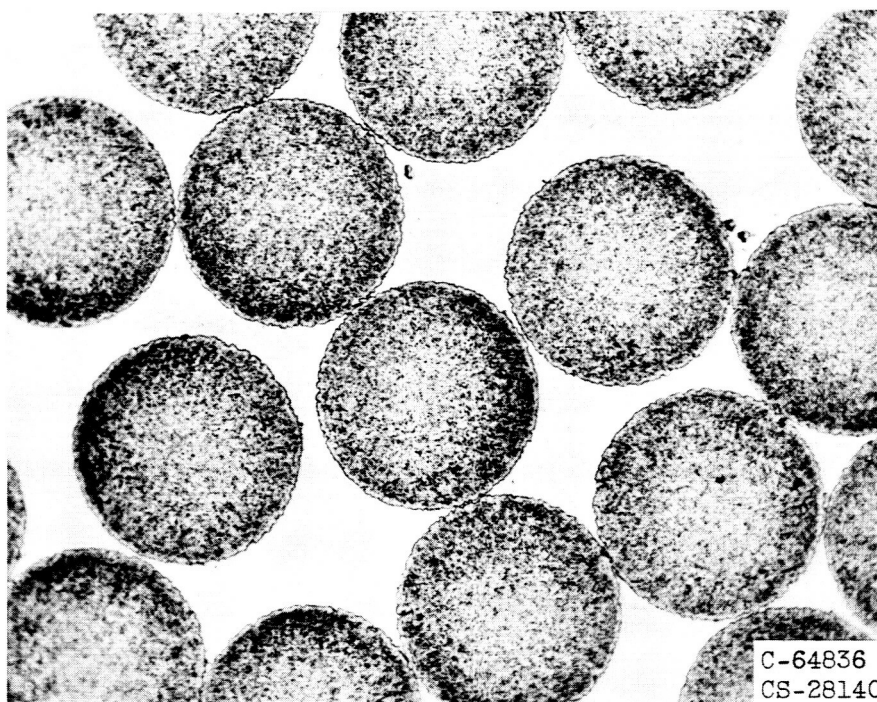


x250

Figure 18. - Copper plus 33 percent zirconium matrix.



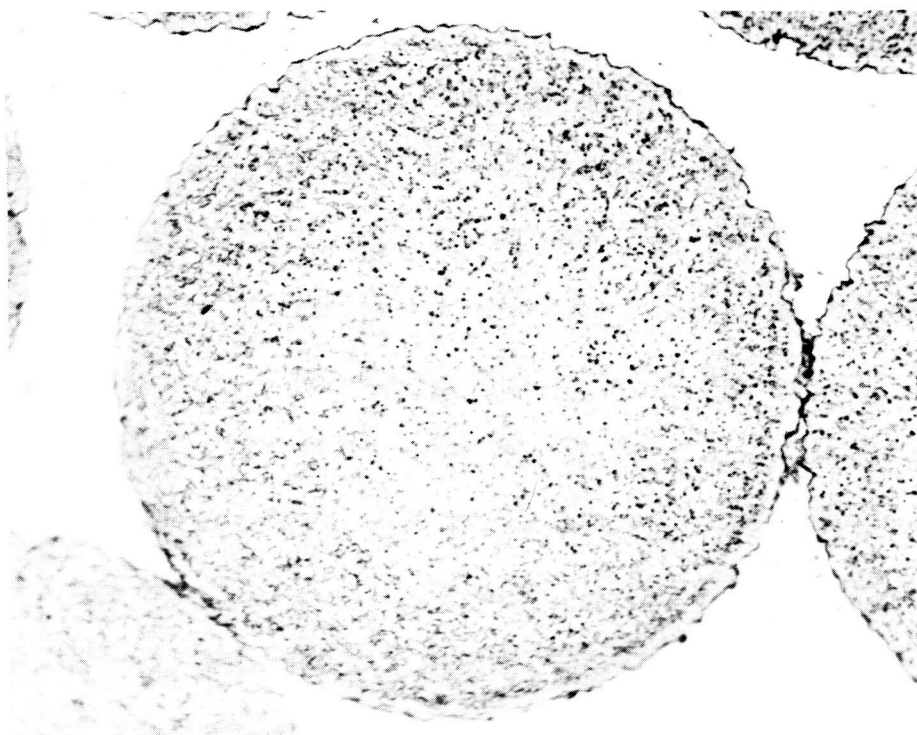
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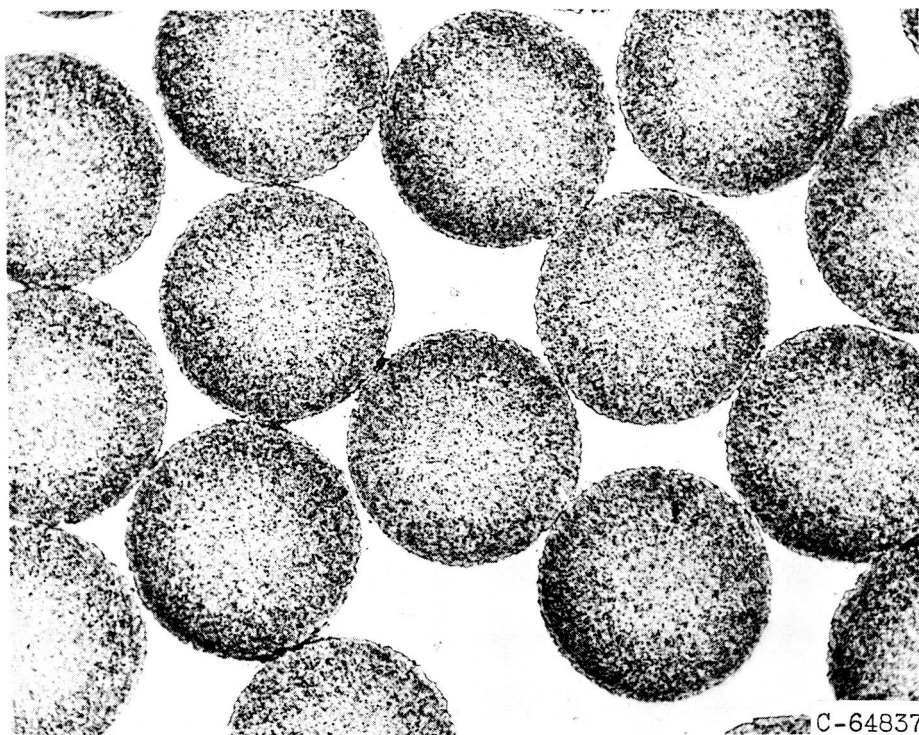
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CS-28140

x250

Figure 19. - Copper plus 1 percent chromium matrix.



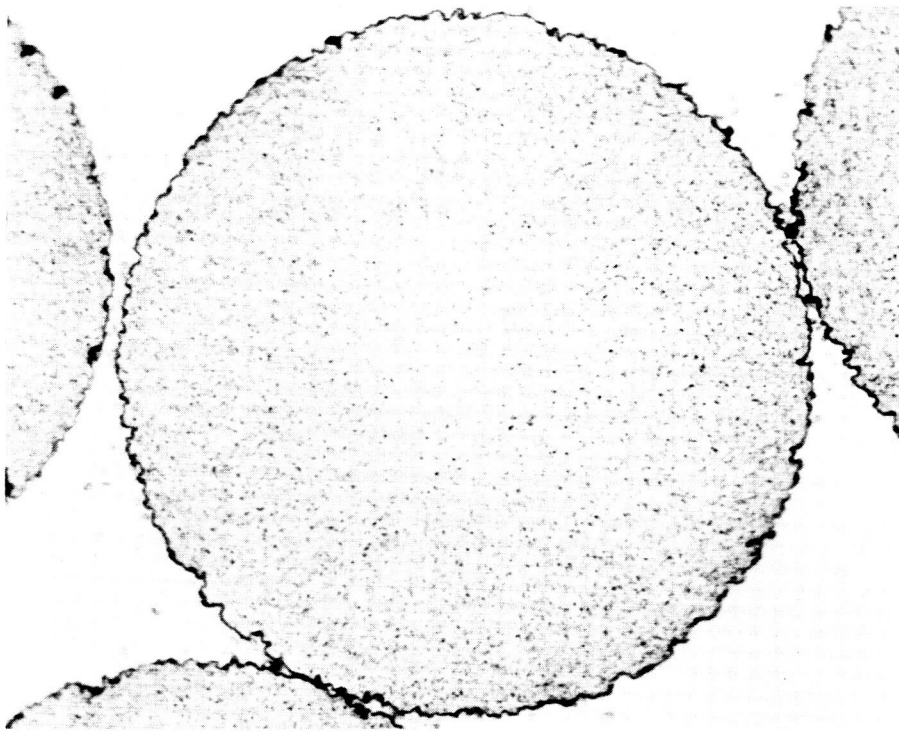
x750



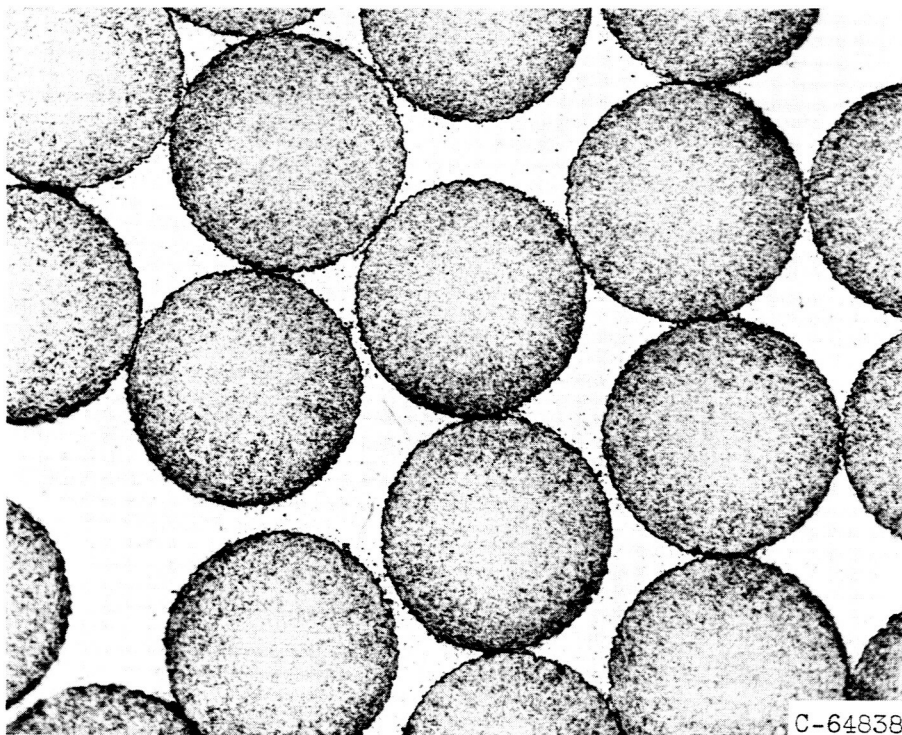
x250

C-64837

Figure 20. - Copper plus 2 percent chromium matrix.



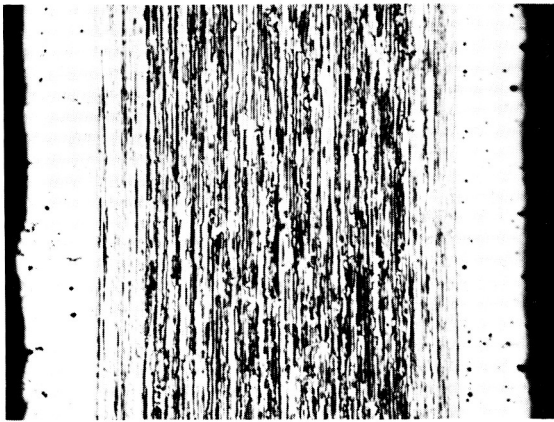
x750



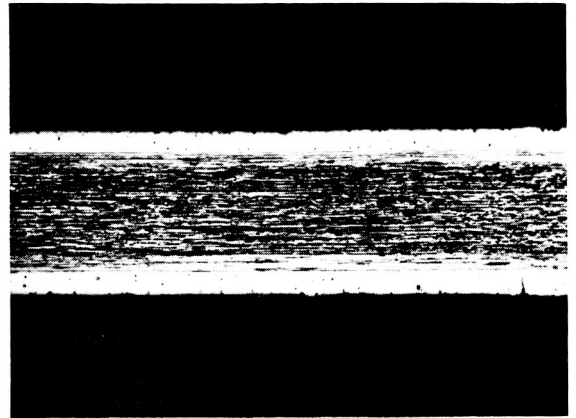
x250

Figure 21. - Copper plus 1 percent niobium matrix.

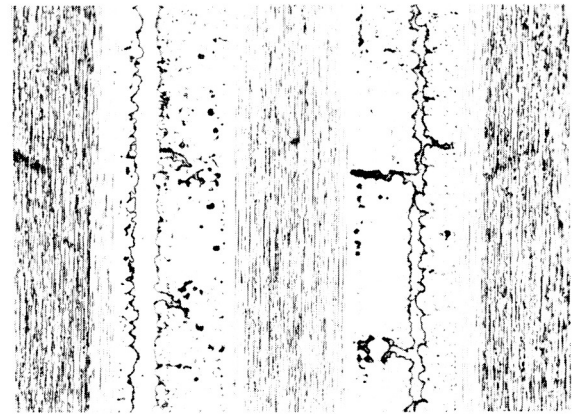
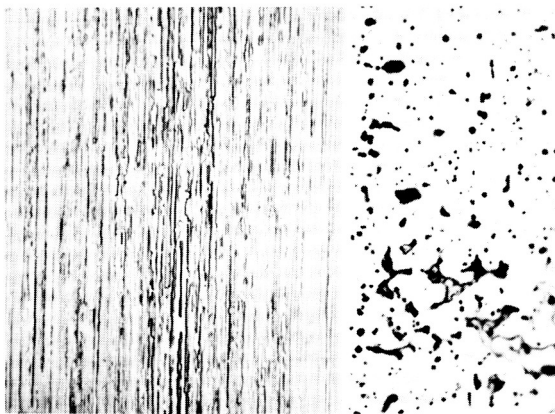
X750



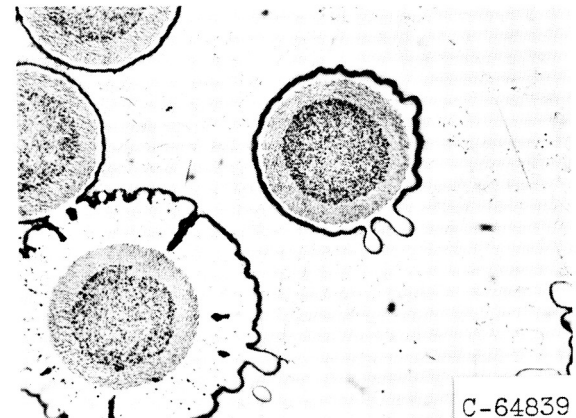
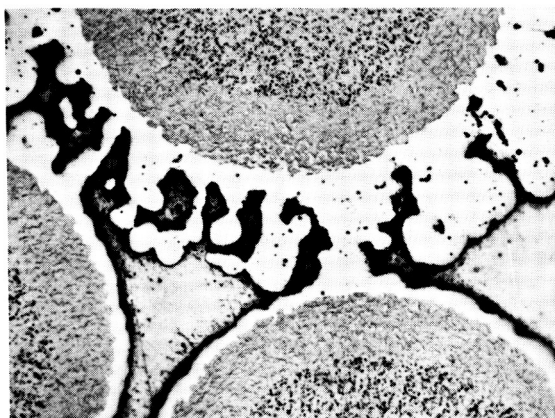
X250



(a) Longitudinal view. Annealed for 1 hour at 500° F, then for 1 hour at 2200° F in vacuum.

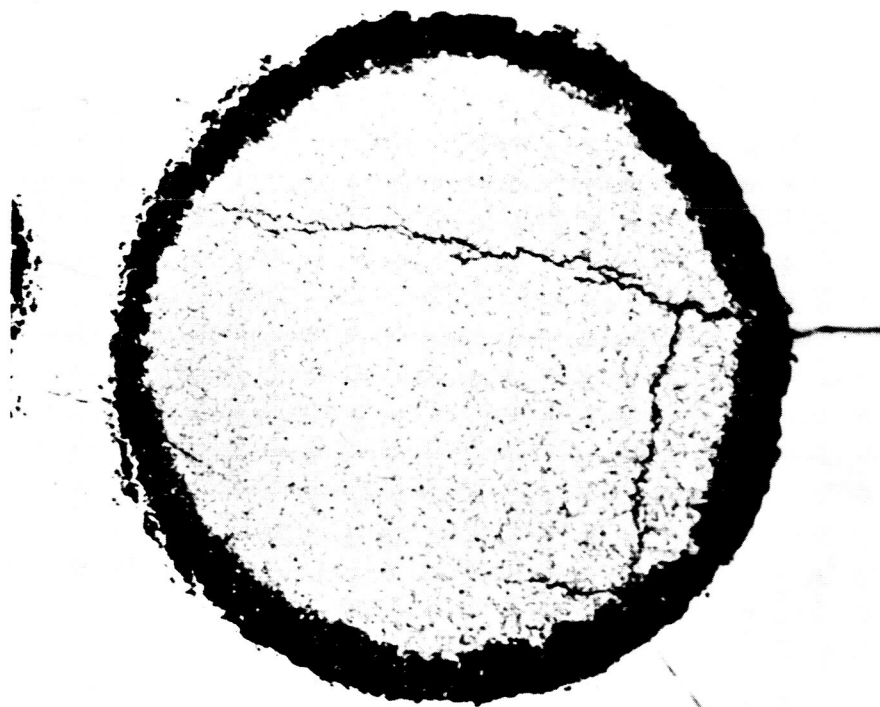


(b) Longitudinal view. Annealed for 1 hour at 500° F, then for 1 hour at 2200° F in vacuum. Copper infiltrated for 1 hour at 2200° F in vacuum.

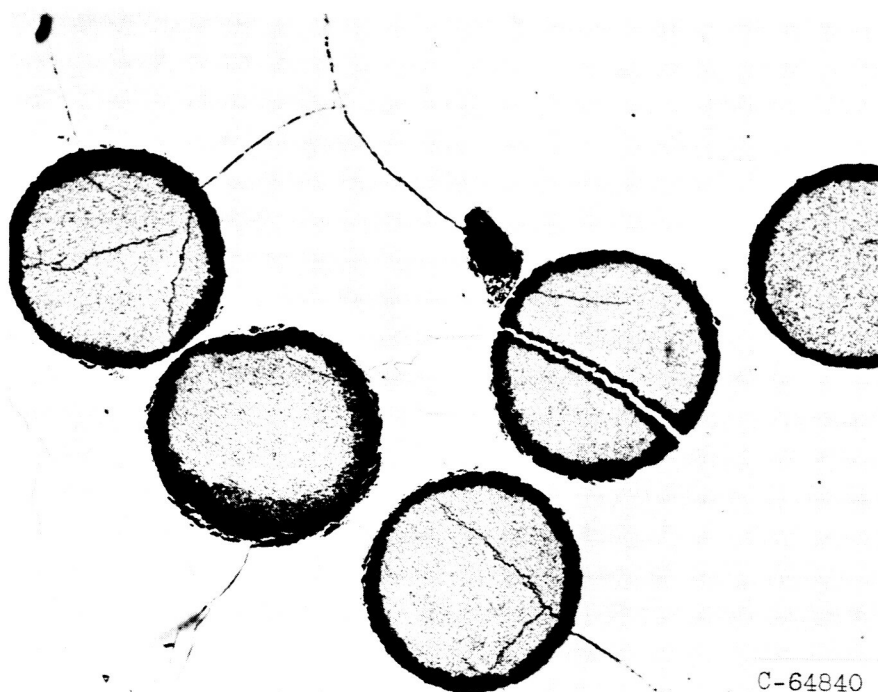


(c) Cross-sectional view. Annealed for 1 hour at 500° F, then for 1 hour at 2200° F in vacuum. Copper infiltrated for 1 hour at 2200° F in vacuum.

Figure 22. - Chromium-plated tungsten-wire study.



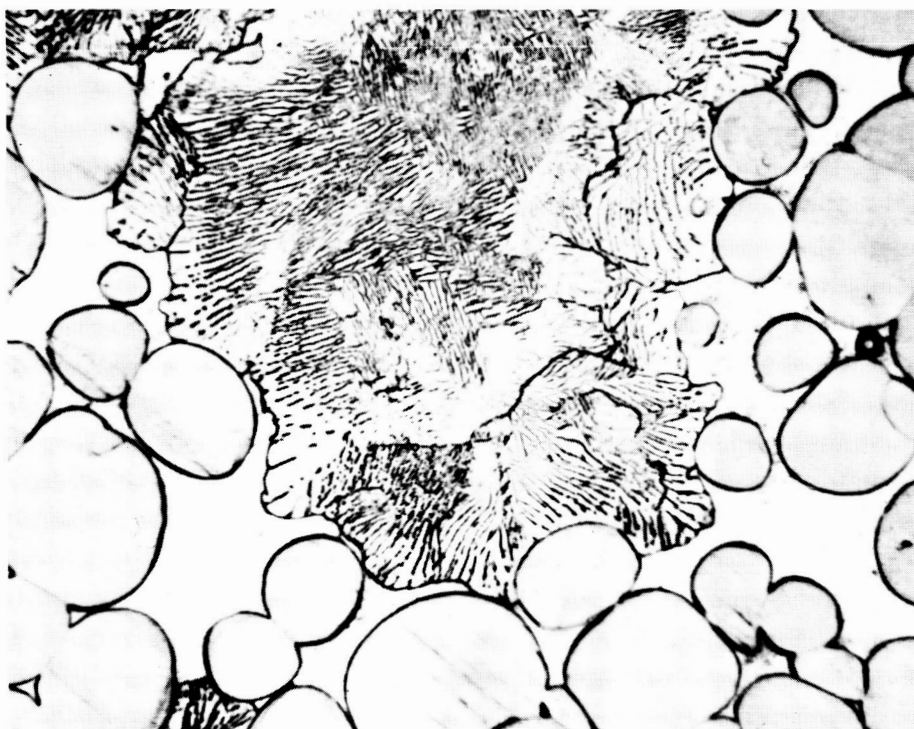
x750



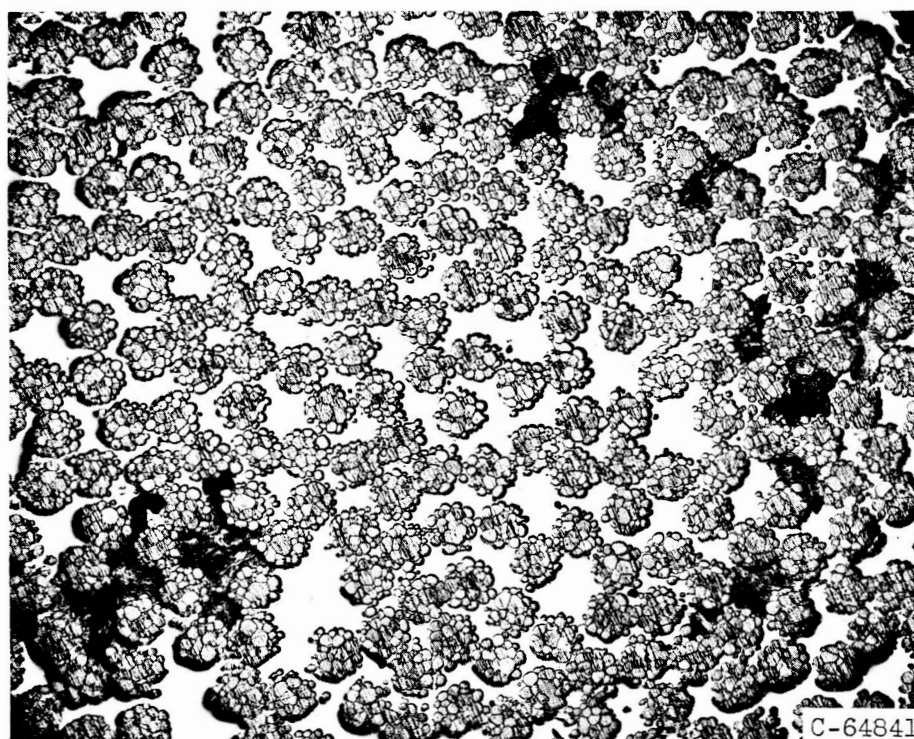
C-64840
CS-28141

x250

Figure 23. - Niobium plus 48 percent nickel matrix.

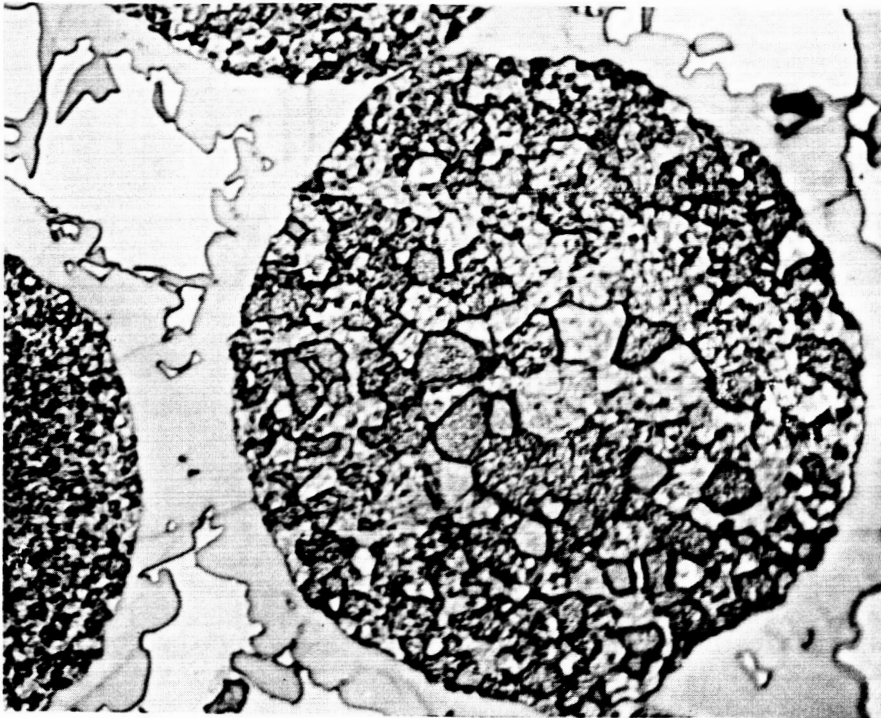


x750

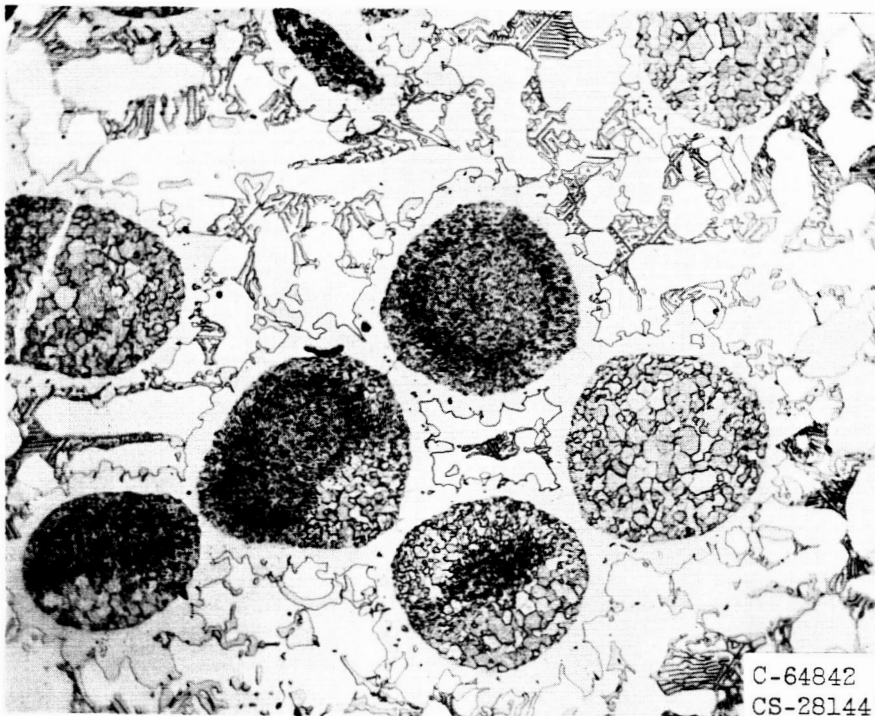


x250

Figure 24. - Nickel plus 30 percent iron matrix.

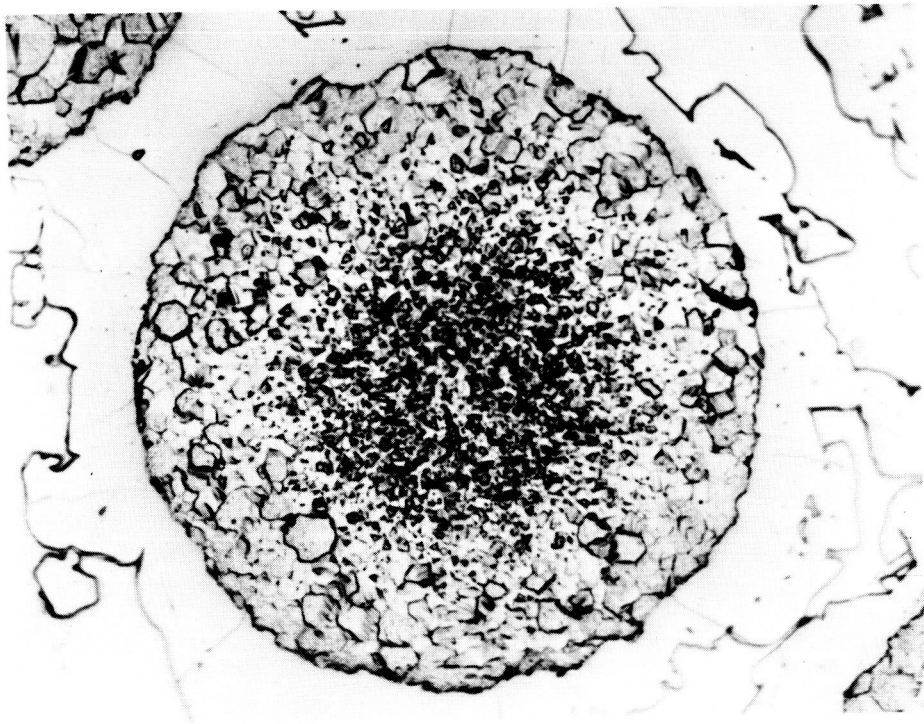


x750

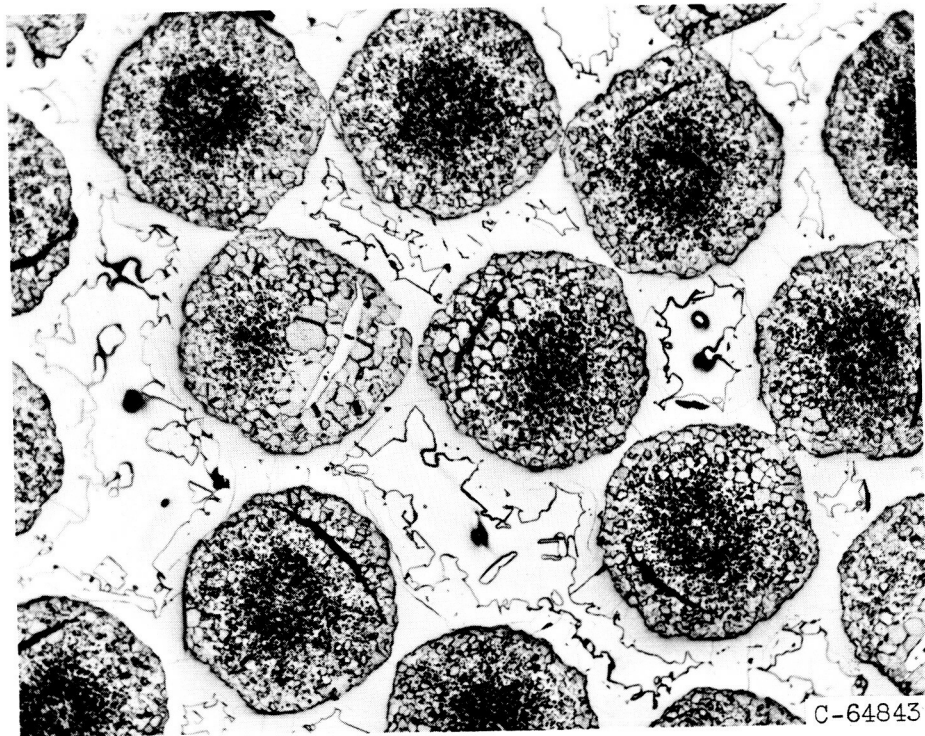


x250

Figure 25. - S-816 matrix.



x750



x250

Figure 26. - L-605 matrix.

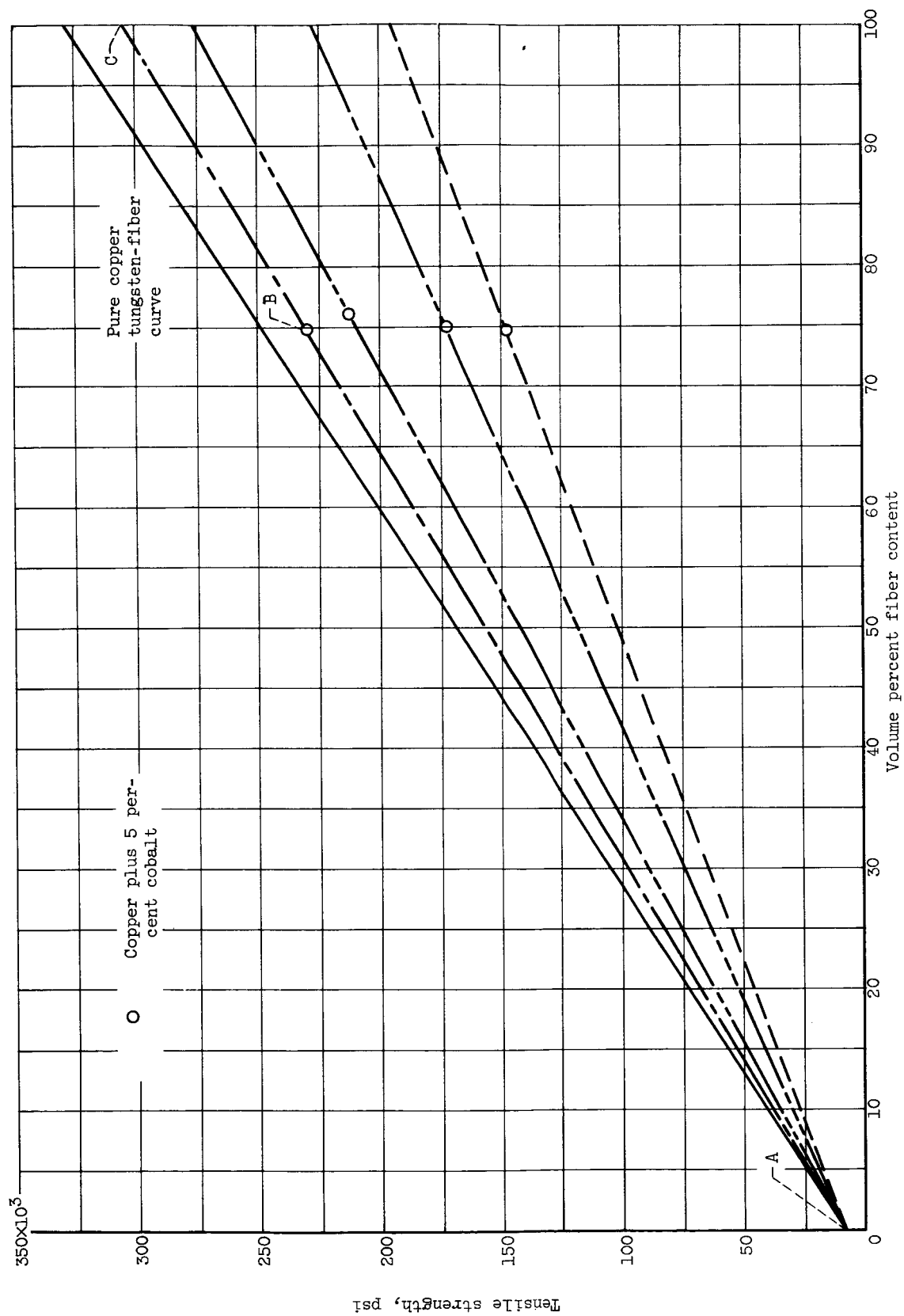


Figure 27. - Strength-composition curve for tungsten-fiber-reinforced - copper plus 5 percent cobalt composites.

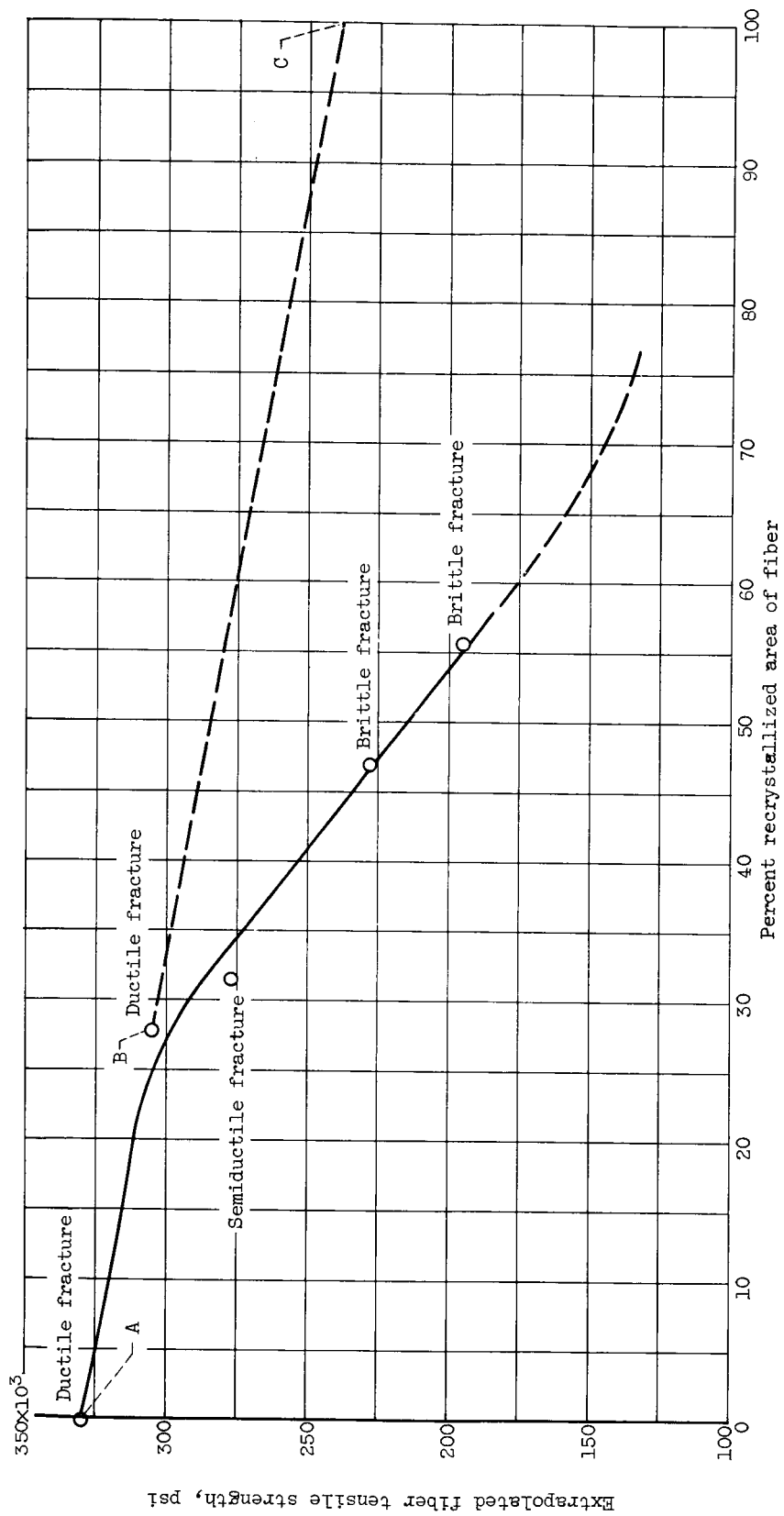


Figure 28. - Fiber tensile strength as a function of percent recrystallized area of fiber for copper plus 5 percent cobalt system.